Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Construction of Polysubstituted Pentafulvenes via Palladium-Catalyzed Deacetylation of Enones

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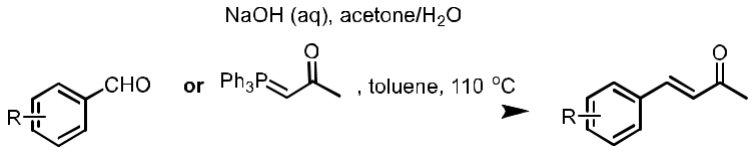
Content

1. General Information

Unless otherwise noted, all reactions were carried out under an N_2 atmosphere in sealed tube with magnetic stirring. All reagents were purchased from commercial suppliers with the highest purity grade, and used directly without further purification. ^{1}H NMR spectra and ^{13}C NMR spectra spectra were recorded on a Bruker 400, 500 and 600 MHz spectrometer in $CDCl_3$ or DMSO-*d*$_6$. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = double of doublets, td = triple doublet, dt = double triplet, and br = broad. EI-double focus magnetic-sector high resolution MS (EI-DFS-HRMS) were recorded on a DFS-Thermofischer instrument at the Center for Mass Spectrometry, Shanghai Institute of Material Medica. Reactions were monitored by thin layer chromatography (TLC) using silica gel plates. Column chromatography was performed on silica gel (200–300 mesh) using a mixture of petroleum ether-ethyl acetate as the eluent.

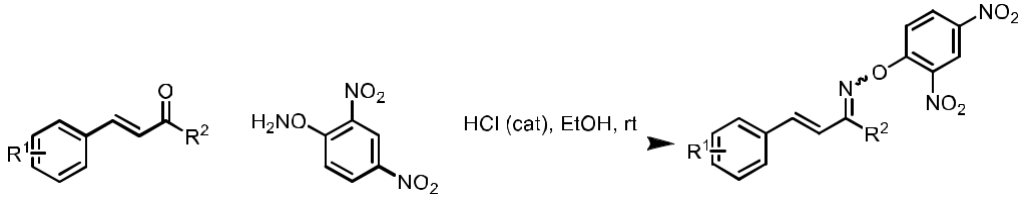
2. General Procedure for the Preparation of Substrates

2.1 Typical procedure for preparation of enone



Enones were commercially available or prepared from corresponding aldehydes according to literature method. [1]

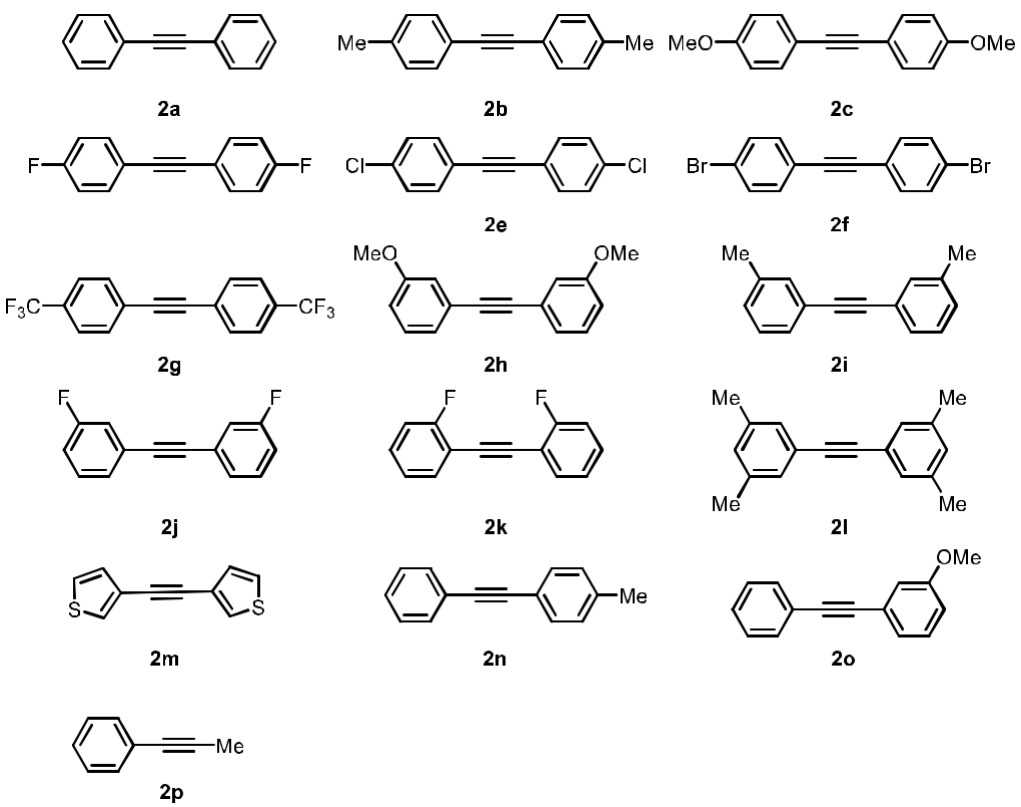
2.2 General procedure for preparation of oxime



Oximes were prepared according to literature method. [2]

To a mixture of enone (3.0 mmol), O-(2,4-dinitrophenyl)hydroxylamine (656.7 mg, 3.3 mmol), EtOH (30 mL) was added conc. HCl (3 drops), and the mixture was stirred at rt for overnight. The reaction mixture was filtered and washed with hexane to give oxime without further purification.

2.3 General procedure for preparation of alkyne

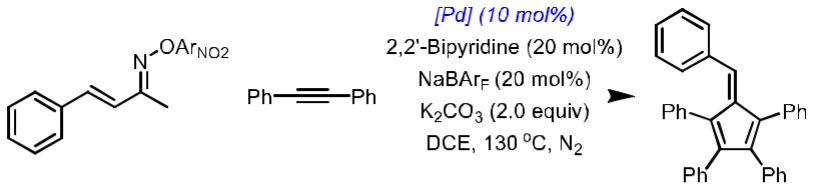


2a-**2o** are known product. **2a**, **2f** and **2o** are commercially available, **2b**-**2e** and **2g**-**2m** were prepared according to literature method.[3a] **2o** and **2p** were prepared according to literature method.[3b]

$Pd(PPh_3)_2Cl_2$ (0.5 mmol, 175 mg), 1,4-bis(diphenylphosphino)butane (DPPB, 1.0 mmol, 0.43 g), aryl bromide (20.0 mmol), 2-butynedionic acid (10.5 mmol, 1.20 g), and DBU (21.0 mmol, 3.20 g) were successively added into DMSO (30.0 mL) in a flame-dried round bottom flask. The mixture was stirred at at 110 °C in a preheated oil bath for 3 h under N_2, and cooled down to room temperature. The reaction mixture was diluted with EtOAc, and washed with saturated NH_4Cl solution for several times. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate) to afford pure alkynes.

3. Optimization of Reaction Condition

Table S1. Screening of [Pd]

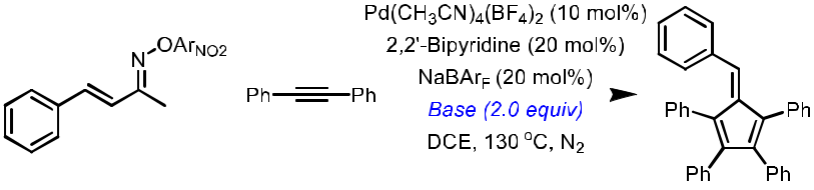


1a, Ar_{NO2} = 2,4-di-$NO_2C_6H_3$ **2a** **3a**

Entry	[Pd]	Yield(%)[a]
1	/	ND
2	$Pd(OAc)_2$	63
3	$PdCl_2$	51
4	$Pd(CH_3CN)_2Cl_2$	48
5	Pd(dppf)Cl_2	27
6	$Pd(CH_3CN)_4(BF_4)_2$	69
7	$Pd(PPh_3)_4$	45
8	$Pd_2(dba)_3$	44

Reaction Condition: **1a** (0.10 mmol), **2a** (0.30 mmol), [Pd] (10 mol%), 2,2'-Bipyridine (20 mol%), NaBAr$_F$ (20 mol%), K_2CO_3 (2.0 equiv), DCE (3.0 mL), 130 °C, N_2 [a] Yield was determined by ^{1}H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

Table S2. Screening of Base

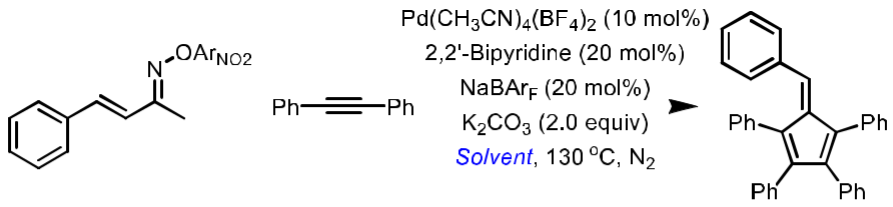


1a, Ar_{NO2} = 2,4-di-$NO_2C_6H_3$ **2a** **3a**

				Base	Yield(%) [a]
				K_3PO_4	62
2	K_2CO_3	69	7	K_2HPO_4	ND
			8	KH_2PO_4	ND
			9	NaOAc	9
5	$KHCO_3$	13			

Reaction Condition: **1a** (0.10 mmol), **2a** (0.30 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (10 mol%), 2,2'-Bipyridine (20 mol%), NaBAr$_F$ (20 mol%), base (0.20 mmol), DCE (3.0 mL), 130 °C, N_2. [a] Yield was determined by ^{1}H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

Table S3. Screening of Solvent

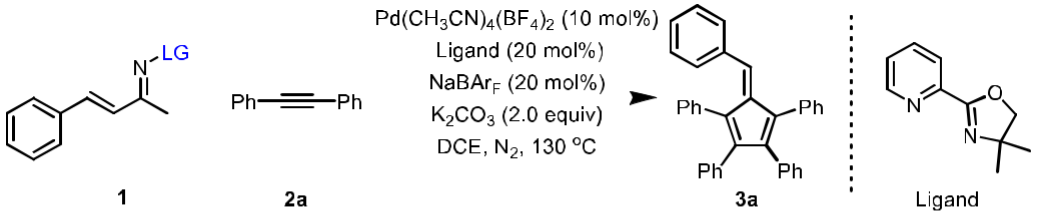


1a, Ar_{NO2} = 2,4-di-$NO_2C_6H_3$ **2a** **3a**

				Solvent	Yield(%)[a]
1	DCE	69	7	toluene	32
			8	MeOH	ND
			9	CH_3CN	ND
			10	DMF	ND
			11	DMSO	ND

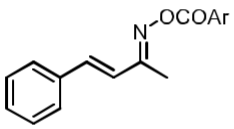
Reaction Condition: **1a** (0.10 mmol), **2a** (0.30 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (10 mol%), 2,2'-Bipyridine (20 mol%), $NaBAr_F$ (20 mol%), K_2CO_3 (0.20 mmol), Solvent (3.0 mL), 130 °C, N_2 [a] Yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

Table S5. Screening of LG

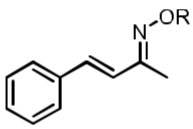


1 **2a** **3a** Ligand

Entry	Substrate	Yield (%)[a]
1	**1-1**	70
2	**1-2**	54
3	**1-3**	42
4	**1-4**	87
5	**1-5**	82
6	**1a**	95(90)[b]
7	**1-6**	ND



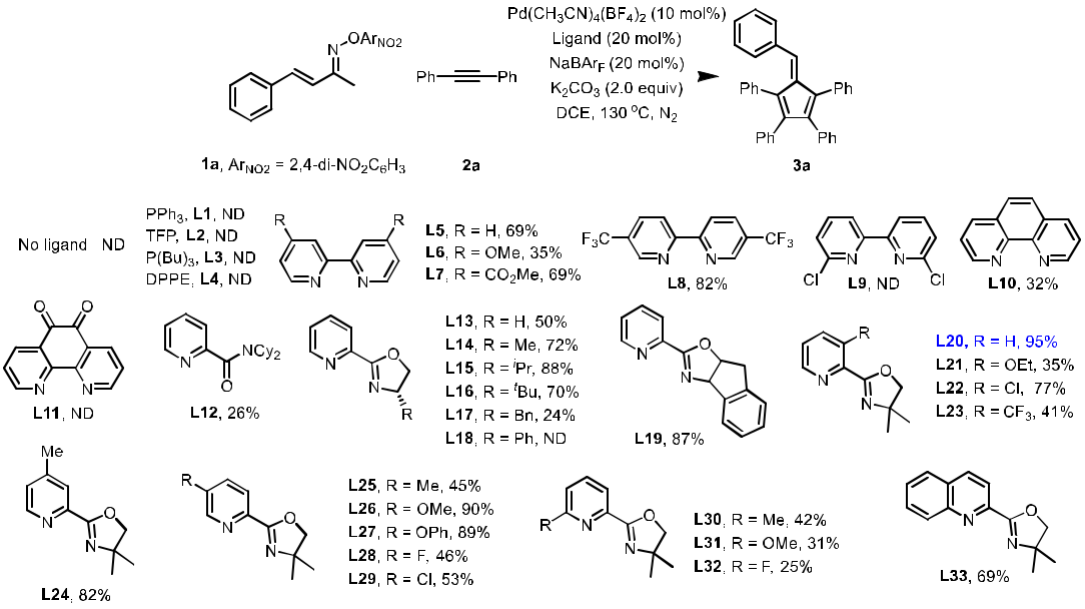
Ar = C_6F_5, **1-1**
= C_6H_5, **1-2**
= *p*-$OMeC_6H_4$, **1-3**
= *p*-$CF_3C_6H_4$, **1-4**



R = *p*-$NO_2C_6H_4$, **1-5**
= 2,4-di-$NO_2C_6H_3$, **1a**
= Me, **1-6**

Reaction conditions: 1 (0.10 mmol), **2a** (0.30 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (10 mol%), Ligand (20 mol%), $NaBAr_F$ (20 mol%), K_2CO_3 (0.20 mmol), DCE, 130 °C, N_2. [a] Yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard. [b] Isolated yield.

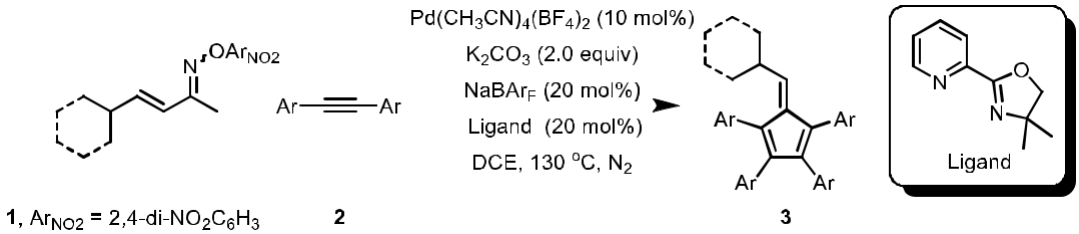
Table S6. Screening of ligand



Reaction Condition: **1a** (0.10 mmol), **2a** (0.30 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (10 mol%), Ligand (20 mol%), $NaBAr_F$ (20 mol%), K_2CO_3 (0.20 mmol), DCE (3.0 mL), 130 °C, N_2 [a] Yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

4. Experimental Procedure

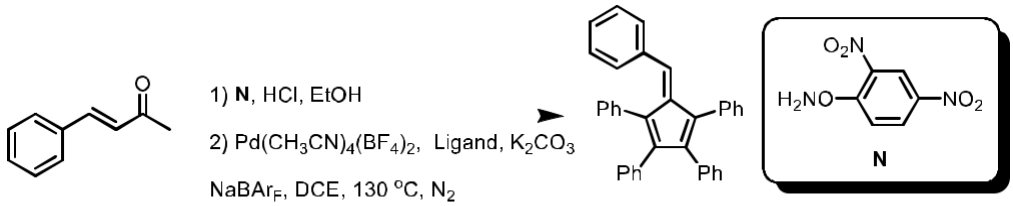
4.1 General procedure for [2+2+1] Cyclization



1, Ar_{NO2} = 2,4-di-$NO_2C_6H_3$ **2** **3**

Oxime **1** (0.1 mmol), diarylacetylene (0.3 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (4.4 mg, 10 mol%), ligand (3.5 mg, 20 mol%), $NaBAr_F$ (17.7 mg, 20 mol%), K_2CO_3 (27.6 mg, 0.2 mmol), DCE (3 mL), and the mixture was stirred at 130 °C in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. The residue was subjected to column chromatography for isolation (gradient eluent: hexane/ethyl acetate) to give product **3**.

4.2 Gram-scale Synthesis and One-pot Protocol

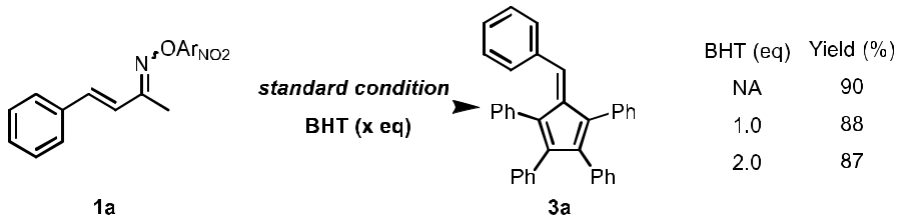


Enone
7.0 mmol, 1.02 g

3a
83% yield, 2.66 g

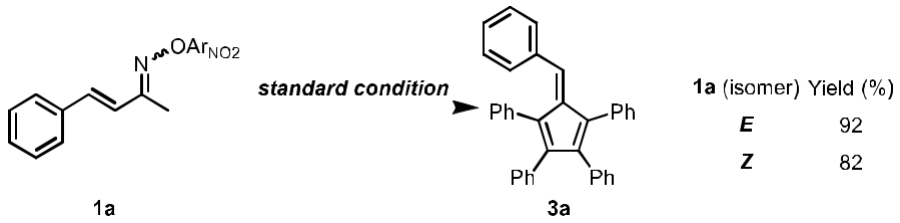
Enone (1.02 g, 7.0 mmol), *O*-(2,4-dinitrophenyl)hydroxylamine (1.39 g, 7.0 mmol), hydrochloric acid in EtOH (40 mL) were added to a 100 mL sealed vials and stirred for 12 h at room temperature. After completion, solvent was removed to give oxime. To the oxime were added diphenyl acetylene (21.0 mmol, 3.74 g), $Pd(CH_3CN)_4(BF_4)_2$ (154 mg, 5 mol%), ligand (245 mg, 20 mol%), $NaBAr_F$ (1.24 g, 20 mol%), K_2CO_3 (1.93 g, 14.0 mmol), DCE (120 mL), and the mixture was stirred at 130 °C in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. The residue was subjected to column chromatography for isolation (gradient eluent: hexane/ethyl acetate) to give product **3a** (2.66 g, 83% yield).

4.3 Radical experiment

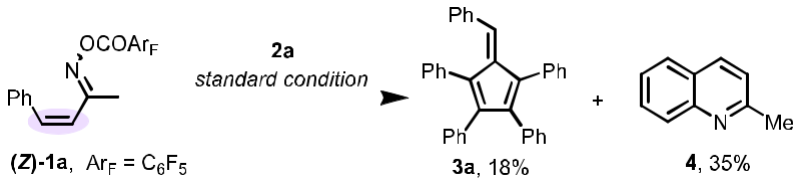


Oxime **1a** (32.7 mg, 0.1 mmol), diphenyl acetylene (53.4 mg, 0.3 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (4.4 mg, 10 mol%), ligand (3.5 mg, 20 mol%), $NaBAr_F$ (17.7 mg, 20 mol%), K_2CO_3 (27.6 mg, 0.2 mmol), BHT, DCE (3 mL), and the mixture was stirred at 130 ^oC in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. Yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

4.4 (*E*/*Z*)-isomer effect experiment

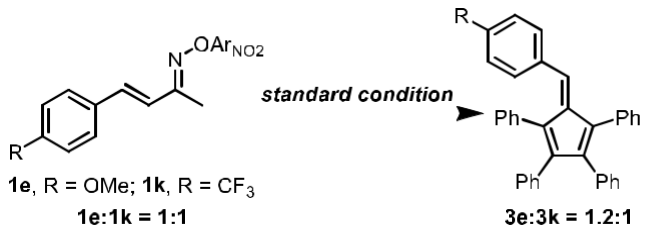


(E/Z)-isomer of Oxime **1a** (32.7 mg, 0.1 mmol), diphenyl acetylene **2a** (53.4 mg, 0.3 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (4.4 mg, 10 mol%), ligand (3.5 mg, 20 mol%), $NaBAr_F$ (17.7 mg, 20 mol%), K_2CO_3 (27.6 mg, 0.2 mmol), DCE (3 mL), and the mixture was stirred at 130 ^oC in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. Yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard.



(Z)-1a (35.5 mg, 0.1 mmol), diphenyl acetylene **2a** (53.4 mg, 0.3 mmol), $Pd(CH_3CN)_4(BF_4)_2$ (4.4 mg, 10 mol%), ligand (3.5 mg, 20 mol%), $NaBAr_F$ (17.7 mg, 20 mol%), K_2CO_3 (27.6 mg, 0.2 mmol), DCE (3 mL), and the mixture was stirred at 130 ^oC in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. The residue was subjected to column chromatography for isolation (gradient eluent: hexane/ethyl acetate) to give product **3a** (8.2 g, 18% yield) and **4** (5.0 mg, 35%).

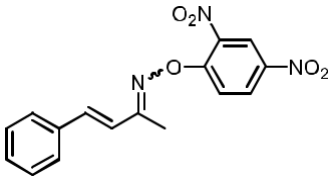
4.5 Competition experiment



Product	Yield (%)
3e	40
3k	33

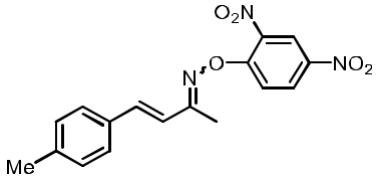
Oxime **1e** (0.1 mmol, 35.7 mg), **1k** (0.1 mmol, 39.5 mg), diphenyl acetylene (0.3 mmol, 53.4 mg), $Pd(CH_3CN)_4(BF_4)_2$ (10 mol%, 4.4 mg), ligand (20 mol%, 3.5 mg), $NaBAr_F$ (20 mol%, 17.7 mg), K_2CO_3 (0.2 mmol, 27.6 mg), DCE (3 mL), and the mixture was stirred at 130 ^oC in oil bath for 12 h. The mixture was filtered through Celite, and evaporated to remove solvent under reduced pressure. Yields of **3e** and **3k** were determined by ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard.

5. Analytical Data



(2E,3E)-4-phenylbut-3-en-2-one O-(2,4-dinitrophenyl) oxime

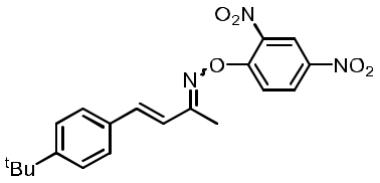
1a: an inseparable mixture of *E/Z* isomers (1.7/1), 1.47 g, 90% yield; yellow solid; m.p. 130–132 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.60H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.44 (m, 1.60H), 8.05 (d, *J* = 9.4 Hz, 0.60H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.78 (d, *J* = 16.4 Hz, 0.60H), 7.62 (d, *J* = 6.8 Hz, 1.20H), 7.54 (d, *J* = 7.2 Hz, 2H), 7.40 (m, 4.80H), 7.18 (d, *J* = 16.4 Hz, 1H), 7.16 (d, *J* = 16.4 Hz, 0.60H), 6.94 (d, *J* = 16.4 Hz, 1H), 2.42 (s, 3H), 2.34 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$)
δ 163.4, 160.3, 157.4, 157.2, 141.1, 140.7, 140.5, 138.3, 135.9, 135.5, 135.3, 135.1, 130.3, 129.6, 129.4, 129.1, 129.0, 128.0, 127.4, 122.8, 122.3, 122.1, 117.2, 117.0, 116.7, 16.4, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{13}N_3O_5$ 327.0850; Found 327.0855.



(2E,3E)-4-(p-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

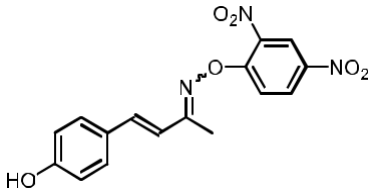
1b: an inseparable mixture of *E/Z* isomers (1.7/1), 1.48 g, 87% yield; yellow solid; m.p. 148–150 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.7 Hz, 0.60H), 8.89 (d, *J* = 2.7 Hz, 1H), 8.43 (m, 1.60H), 8.04 (d, *J* = 9.4 Hz, 0.60H), 8.03 (d, *J* = 9.3 Hz, 1H), 7.72 (d, *J* = 16.4 Hz, 0.60H), 7.50 (d, *J* = 8.1 Hz, 1.20H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.22 (m, 3.20H), 7.14 (d, *J* = 16.4 Hz, 1H), 7.13 (d, *J* = 16.4 Hz, 0.60H), 6.89 (d, *J* = 16.4 Hz, 1H), 2.41 (s, 3H), 2.39 (s, 4.80H), 2.32 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 160.4, 157.5, 157.3, 141.2, 140.8, 140.7, 140.4, 134.0, 138.3, 136.0, 135.6,

132.6, 132.4, 129.8, 129.7, 129.6, 129.4, 128.0, 127.3, 122.3, 122.1, 121.8, 117.3, 117.1, 115.7, 21.5, 21.4, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_5$ 341.1006; Found 341.1008.



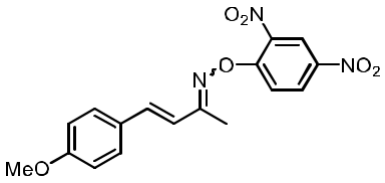
(2E,3E)-4-(4-(tert-butyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1c: an inseparable mixture of *E/Z* isomers (1.7/1), 1.53 g, 80% yield; yellow solid; m.p. 169–171 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.60H), 8.90 (d, *J* = 2.7 Hz, 1.20H), 8.47–8.43 (m, 1.60H), 8.05 (d, *J* = 9.3 Hz, 0.60H), 8.04 (d, *J* = 9.3 Hz, 1H), 7.74 (d, *J* = 16.3 Hz, 0.60H), 7.56 (d, *J* = 8.5 Hz, 1.20H), 7.51–7.39 (m, 5.20H), 7.16 (d, *J* = 16.4 Hz, 1H), 7.15 (d, *J* = 16.4 Hz, 0.60H), 6.90 (d, *J* = 16.4 Hz, 1H), 2.41 (s, 3H), 2.32 (s, 1.80H), 1.35 (s, 14.40H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 160.5, 157.5, 157.3, 153.9, 153.1, 141.0, 140.7, 140.4, 138.2, 136.0, 135.6, 132.6, 132.4, 129.6, 129.4, 127.9, 127.2, 126.1, 126.0, 122.3, 122.1, 121.9, 117.3, 117.0, 116.0, 34.94, 34.87, 31.20, 31.17, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{20}H_{21}N_3O_5$ 383.1476; Found 383.1480.



(2E,3E)-4-(4-hydroxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

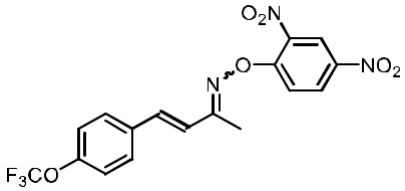
1d: an inseparable mixture of *E/Z* isomers (2/1), 1.11 g, 65% yield; yellow solid; m.p. 176–177 °C. **^{1}H NMR** (500 MHz, DMSO-*d*6) δ 10.10 (s, 0.50H), 9.92 (s, 1H), 8.89–8.84 (m, 1.50H), 8.64–8.56 (m, 1.50H), 8.00 (d, *J* = 9.4 Hz, 1.50H), 8.60–8.49 (m, 3H), 7.41 (d, *J* = 14.5 Hz, 0.50H), 7.35 (d, *J* = 16.2 Hz, 1H), 6.92–6.76 (m, 4.50H), 2.37 (s, 3H), 2.31 (s, 1.50H). **^{13}C NMR** (125 MHz, DMSO) δ 164.1, 161.0, 159.8, 158.9, 156.5, 156.2, 141.8, 140.4, 140.1, 138.9, 135.8, 135.4, 130.2, 130.0, 129.7, 129.2, 126.4, 126.1, 122.0, 121.9, 118.6, 117.0, 116.9, 116.0, 115.7, 112.4, 16.1, 11.7. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{13}N_3O_6$ 343.0799; Found 343.0806.



(2E,3E)-4-(4-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1e: an inseparable mixture of *E/Z* isomers (1.8/1), 1.43 g, 80% yield; yellow solid; m.p. 149–151 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.93 (d, *J* = 2.7 Hz, 0.55H), 8.87 (d, *J* = 2.7 Hz, 1H), 8.42 (m, 1.55H), 8.03 (d, *J* = 9.4 Hz, 0.55H), 8.02 (d, *J* = 9.3 Hz, 1H), 7.61 (d, *J* = 16.4 Hz, 0.55H), 7.55 (d, *J* = 8.7 Hz, 1.10H), 7.46 (d, *J* = 8.7 Hz, 2H), 7.10 (d, *J* = 16.4 Hz, 1H), 7.09 (d, *J* = 16.4 Hz, 0.55H), 6.96 – 6.90 (m, 3.10H), 6.79 (d, *J* = 16.4 Hz, 1H), 3.85 (s, 4.63H), 2.39 (s, 3H), 2.30 (s, 1.64H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 161.4, 160.8, 160.5, 157.6, 157.3, 140.7, 140.6, 140.4, 137.9, 135.9, 135.5, 129.7, 129.6, 129.4, 128.8, 128.1, 127.8, 122.3, 122.1, 120.4, 117.2, 117.0, 114.5,

114.4, 114.4, 55.4, 55.4, 16.4, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_6$ 357.0954; Found 357.0955.

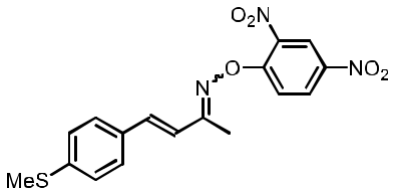


(2E,3E)-4-(4-(trifluoromethoxy)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1f: an inseparable mixture of *E/Z* isomers (10/1), 1.40 g, 68% yield; yellow solid; m.p. 147–149 °C.

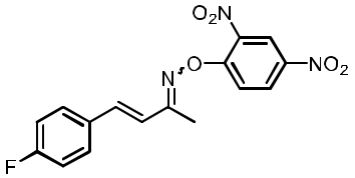
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.7 Hz, 0.10H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.46 (dd, *J* = 9.3, 2.7 Hz, 1.10H), 8.05 (d, *J* = 9.4 Hz, 0.10H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.76 (d, *J* = 16.5 Hz, 0.10H), 7.65 (d, *J* = 8.6 Hz, 0.20H), 7.56 (d, *J* = 8.6 Hz, 2H), 7.25 (d, *J* = 7.0 Hz, 2.20H), 7.16 (d, *J* = 16.4 Hz, 1H), 7.18 (d, *J* = 16.4 Hz, 0.10H), 6.92 (d, *J* = 16.4 Hz, 1H), 2.42 (s, 3H), 2.34 (s, 0.30H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 163.1, 157.1, 149.9, 140.9, 136.5, 136.1, 134.0, 129.4, 128.7, 128.1 (q, *J* = 286.2 Hz), 123.9, 122.1, 121.3, 117.2, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{12}F_3N_3O_6$ 411.0673; Found 411.0677.



(2E,3E)-4-(4-(methylthio)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

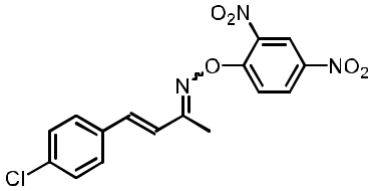
1g: an inseparable mixture of *E/Z* isomers (2/1), 1.37 g, 80% yield; yellow solid; m.p. 150–152 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.8 Hz, 0.50H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.47–8.43 (m, 1.5H), 8.05 (d, *J* = 9.4 Hz, 0.50H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.73 (d, *J* = 16.4 Hz, 0.50H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.29–7.23 (m, 3H), 7.12 (d, *J* = 16.5 Hz, 1H), 7.14 (d, *J* = 16.5 Hz, 0.50H), 6.89 (d, *J* = 16.4 Hz, 1H), 2.52 (s, 4.50H), 2.41 (s, 3H), 2.32 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.5, 160.3, 157.5, 157.3, 141.0, 140.8, 140.5, 137.7, 136.0, 131.9, 131.6, 129.6, 129.4, 128.4, 127.7, 126.2, 126.1, 122.4, 122.1, 121.9, 117.3, 117.1, 115.8, 77.3, 77.0, 76.8, 16.4, 15.3, 15.2, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_5S$ 373.0727; Found 373.0727.



(2E,3E)-4-(4-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

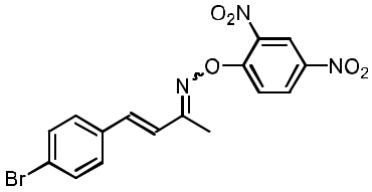
1h: an inseparable mixture of *E/Z* isomers (1/1), 1.29 g, 76% yield; yellow solid; m.p. 163–165 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.8 Hz, 1H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.46 (dd, *J* = 2.8, 1.0 Hz, 1H), 8.44 (dd, *J* = 2.8, 1.1 Hz, 1H), 8.05 (d, *J* = 9.4 Hz, 1H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.71 (d, *J* = 16.4 Hz, 1H), 7.61 (m, 2H), 7.52 (m, 2H), 7.17–7.08 (m, 6H), 6.86 (d, *J* = 16.4 Hz, 1H), 2.42 (s, 3H), 2.33 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 165.5 (d, *J* = 207.9 Hz), 165.1 (d, *J* = 192.0 Hz), 163.9, 161.6, 158.8, 158.6, 142.3, 142.0, 141.1, 138.4, 137.5, 137.1, 133.0 (d, *J* = 3.1 Hz), 132.8 (d, *J* = 3.6 Hz), 131.3 (d, *J* = 8.5 Hz), 131.1, 130.8, 130.5 (d, *J* = 8.4 Hz), 124.1 (d, *J* = 2.1

Hz), 123.8, 123.5, 118.7, 118.5, 118.0 (d, J = 2.1 Hz), 117.7 (d, J = 21.9 Hz), 117.5 (d, J = 22.7 Hz), 17.8, 17.8, 13.6, 13.6. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}FN_3O_5$ 345.0756; Found 345.0759.



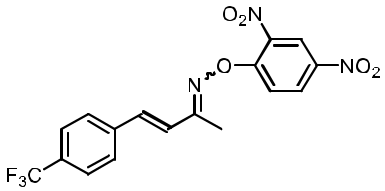
(2E,3E)-4-(4-chlorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1i: an inseparable mixture of *E/Z* isomers (1.3/1), 1.53 g, 85% yield; yellow solid; m.p. 156–158 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.96 (d, J = 2.7 Hz, 0.75H), 8.91 (d, J = 2.7 Hz, 1H), 8.49–8.45 (m, 1.75H), 8.05 (d, J = 9.4 Hz, 0.75H), 8.02 (d, J = 9.4 Hz, 1H), 7.75 (d, J = 16.5 Hz, 0.75H), 7.55 (d, J = 8.5 Hz, 1.50H), 7.47 (d, J = 8.5 Hz, 2H), 7.42–7.35 (m, 3.50H), 7.12 (d, J = 16.4 Hz, 1H), 7.10 (d, J = 16.4 Hz, 0.75H), 6.91 (d, J = 16.5 Hz, 1H), 2.41 (s, 3H), 2.33 (s, 2.25H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.2, 160.0, 157.3, 157.1, 140.9, 140.6, 139.6, 136.8, 136.2, 136.1, 135.6, 135.4, 133.9, 133.6, 129.7, 129.4, 129.4, 129.2, 129.2, 128.5, 123.5, 122.4, 122.1, 117.3, 117.2, 117.0, 16.42, 12.19. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}ClN_3O_5$ 361.0460; Found 361.0470.



(2E,3E)-4-(4-bromophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

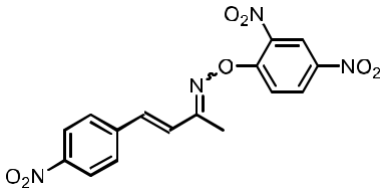
1j: an inseparable mixture of *E/Z* isomers (1.5/1), 1.58 g, 78% yield; yellow solid; m.p. 160–162 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, J = 2.7 Hz, 0.65H), 8.90 (d, J = 2.7 Hz, 1H), 8.47–7.43 (m, 1.65H), 8.04 (d, J = 9.4 Hz, 0.65H), 8.02 (d, J = 9.4 Hz, 1H), 7.76 (d, J = 16.5 Hz, 0.65H), 7.67–7.51 (m, 3.30H), 7.47 (d, J = 8.5 Hz, 1.30H), 7.40 (d, J = 8.5 Hz, 2H), 7.10 (d, J = 16.4 Hz, 1H), 7.08 (d, J = 16.4 Hz, 0.65H), 6.92 (d, J = 16.4 Hz, 1H), 2.41 (s, 3H), 2.33 (s, 1.95H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.2, 160.0, 157.3, 157.1, 140.9, 140.6, 139.6, 136.9, 136.1, 135.6, 134.3, 134.0, 132.4, 132.2, 129.7, 129.4, 129.4, 128.8, 124.6, 123.7, 123.6, 122.4, 122.1, 117.4, 117.2, 117.1, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}BrN_3O_5$ 404.9955; Found 404.9947.



(2E,3E)-4-(4-(trifluoromethyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1k: an inseparable mixture of *E/Z* isomers (1.8/1), 1.58 g, 80% yield; yellow solid; m.p. 165–167 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, J = 2.7 Hz, 0.55H), 8.90 (d, J = 2.7 Hz, 1H), 8.45 (d, J = 9.2 Hz, 1.55H), 8.07–8.02 (m, 1.55H), 7.85 (d, J = 16.5 Hz, 0.55H), 7.76–7.61 (m, 6.20H), 7.20 (d, J = 16.5 Hz, 1H), 7.18 (d, J = 16.5 Hz, 0.55H), 7.02 (d, J = 16.4 Hz, 1H), 2.44 (s, 3H), 2.36 (s, 1.65H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.0, 159.8, 157.2, 157.0, 141.0, 140.7, 139.1, 138.7, 138.5, 136.4, 136.2, 135.7, 132.2 (q, J = 31.5 Hz), 131.1 (q, J = 32.8 Hz), 129.7, 129.4, 128.2, 127.5, 126.1 (q, J = 4.0 Hz), 125.9 (q, J = 4.1 Hz), 125.5, 123.9 (q, J = 272.2 Hz), 123.8 (q, J = 272.2 Hz), 122.4, 122.1, 119.2, 117.2, 117.0, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{12}F_3N_3O_5$ 395.0724;

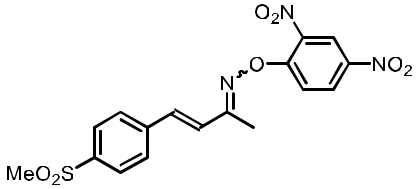
Found 395.0730.



(2E,3E)-4-(4-nitrophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1l: an inseparable mixture of *E/Z* isomers (1/1.3), 1.40 g, 75% yield; yellow solid; m.p. 189–191 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.98 (d, *J* = 2.8 Hz, 1.30H), 8.93 (d, *J* = 2.8 Hz, 1H), 8.48 (dd, *J* = 9.4, 2.7 Hz, 2.30H), 8.33–8.25 (m, 4.60H), 8.09–8.02 (m, 2.30H), 7.93 (d, *J* = 16.5 Hz, 1.30H), 7.78 (d, *J* = 8.4 Hz, 2.60H), 7.69 (d, *J* = 8.6 Hz, 2H), 7.26–7.17 (m, 2.30H), 7.09 (d, *J* = 16.5 Hz, 1H), 2.46 (s, 3H), 2.38 (s, 3.90H). **^{13}C NMR** (125 MHz, DMSO-*d*$_6$) δ 163.6, 160.5, 156.1, 155.9, 147.9, 147.4, 147.2, 141.9, 141.6, 140.8, 140.5, 139.2, 136.4, 136.0, 135.6, 130.2, 130.1, 128.8, 128.6,

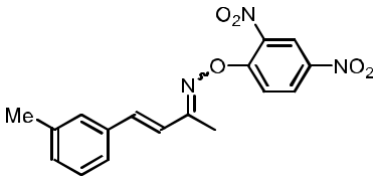
126.8, 124.3, 124.0, 122.1, 121.9, 119.7, 117.1, 16.3, 11.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}N_4O_7$ 372.0701; Found 372.0701.



(2E,3E)-4-(4-(methylsulfonyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

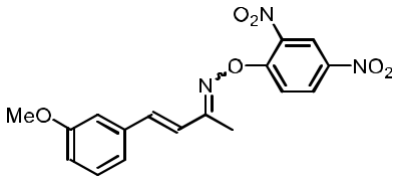
1m: an inseparable mixture of *E/Z* isomers (10/1), 1.42 g, 70% yield; yellow solid; m.p. 203–204 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.97 (d, *J* = 2.7 Hz, 0.10H), 8.92 (d, *J* = 2.8 Hz, 1H), 8.47 (dd, *J* = 9.4, 2.8 Hz, 1.10H), 8.05–7.95 (m, 3.30H), 7.90 (d, *J* = 16.6 Hz, 0.10H), 7.80 (d, *J* = 8.2 Hz, 0.20H), 7.72 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 16.4 Hz, 1H), 7.19 (d, *J* = 16.4 Hz, 0.10H), 7.07 (d, *J* = 16.4 Hz, 1H), 3.09 (s, 3H), 3.08 (s, 0.30H), 2.45 (s, 3H), 2.37 (s, 0.30H). **^{13}C NMR** (125 MHz, DMSO-*d*$_6$) δ 163.7, 160.6, 156.2, 155.9, 141.6, 140.8, 140.7, 140.5, 140.2, 140.0, 139.7, 136.9, 135.9, 135.5, 130.2, 130.1, 128.4, 128.2, 127.7, 127.5, 125.8, 122.1, 121.9, 119.0, 117.1, 117.0, 43.4, 43.3, 16.3, 11.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_7S$ 405.0625; Found 405.0631.



(2E,3E)-4-(m-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

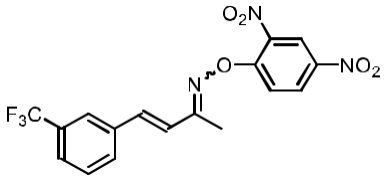
1n: an inseparable mixture of *E/Z* isomers (1.7/1), 1.45 g, 85% yield; yellow solid; m.p. 129–131 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.7 Hz, 0.60H), 8.88 (d, *J* = 2.7 Hz, 1H), 8.46–8.42 (m, 1.60H), 8.03 (d, J = 9.4 Hz, 0.60H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.74 (d, *J* = 16.4 Hz, 0.60H), 7.43 (d, *J* = 7.7 Hz, 0.60H), 7.39 (s, 0.60H), 7.36–7.27 (m, 3.60H), 7.21 (d, *J* = 7.5 Hz, 0.60H), 7.18 (d, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 16.4 Hz, 1H), 7.12 (d, *J* = 16.4 Hz, 0.60H), 6.92 (d, *J* = 16.4 Hz, 1H), 2.41 (s, 3H), 2.40 (s, 1.80H), 2.39 (s, 3H), 2.32 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.5, 160.4, 157.5, 157.2, 141.3, 140.8, 140.5, 138.8, 138.7, 138.4, 136.0, 135.6, 135.3, 135.0, 131.2, 130.5,
129.6, 129.4, 129.0, 128.94, 128.87, 128.0, 125.0, 124.6, 122.6, 122.3, 122.1, 117.2, 117.1, 116.5, 21.40, 21.38, 16.4, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_5$ 341.1006; Found 341.1015.



(2E,3E)-4-(3-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1o: an inseparable mixture of *E/Z* isomers (1.7/1), 1.43 g, 80% yield; yellow solid; m.p. 122–124 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.60H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.48–8.43 (m, 1.60H), 8.05 (d, *J* = 9.4 Hz, 0.60H), 8.04 (d, *J* = 9.3 Hz, 1H), 7.79 (d, *J* = 16.4 Hz, 0.60H), 7.36–7.29 (m, 1.60H), 7.19 (d, *J* = 7.6 Hz, 0.60H), 7.18–7.10 (m, 3.20H), 7.08–7.03 (m, 1H), 6.97–6.89 (m, 2.60H), 3.89 (s, 1.80H), 3.86 (s, 3H), 2.42 (s, 3H), 2.33 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.4, 160.3, 160.0, 157.4, 157.2, 140.9, 140.8, 140.5, 138.2, 136.7, 136.5, 136.1, 130.1, 123.0, 129.6, 129.4, 123.2, 122.4, 122.1, 120.8, 120.1, 117.3, 117.1, 117.0, 116.6, 115.3, 112.4, 112.3, 55.3, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_6$ 357.0955; Found 357.0959.

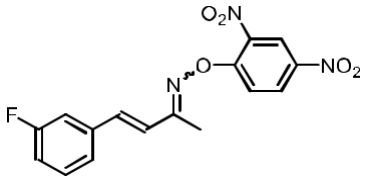


(2E,3E)-4-(3-(trifluoromethyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1p: an inseparable mixture of *E/Z* isomers (3.3/1), 1.58 g, 80% yield; yellow solid; m.p. 130–132 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.01 (d, *J* = 2.7 Hz, 0.30H), 8.96 (d, *J* = 2.7 Hz, 1H), 8.57–8.39 (m, 1.30H), 8.09 (d, *J* = 9.4 Hz, 0.30H), 8.08 (d, *J* = 9.4 Hz, 1H), 7.93 (d, *J* = 7.8 Hz, 0.30H), 7.89 (d, *J* = 16.5 Hz, 0.30H), 7.84–7.80 (m, 1.30H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.71–7.53 (m, 2.60H), 7.24 (d, *J* = 16.5 Hz, 1H), 7.22 (d, *J* = 16.5 Hz, 0.30), 7.05 (d, *J* = 16.5 Hz, 1H), 2.47 (s, 3H), 2.39 (s, 0.90H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 159.8, 157.2, 157.0, 141.0, 140.7, 139.2, 136.5, 136.14, 136.10, 135.9, 135.7, 131.54 (q, *J* = 32.7 Hz), 131.50 (q, *J* = 32.6 Hz), 130.4, 130.0, 129.7, 129.6, 129.5, 129.4, 126.7 (q, *J* = 3.8 Hz), 126.0 (d, *J* = 4.0 Hz), 125.5 (d, *J* = 3.9 Hz), 124.9, 123.94 (q, *J* = 3.8 Hz), 123.86 (q, *J* = 272.16 Hz), 123.8 (q, *J* = 272.16 Hz) 122.3, 122.1, 118.6, 117.2, 117.1, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{12}F_3N_3O_5$ 395.0724; Found 395.0748.

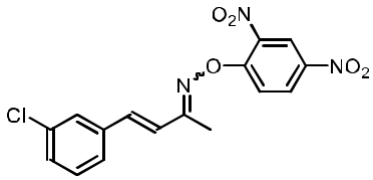


(2E,3E)-4-(3-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1q: an inseparable mixture of *E/Z* isomers (2/1), 1.21 g, 70% yield; yellow solid; m.p. 138–140 °C.

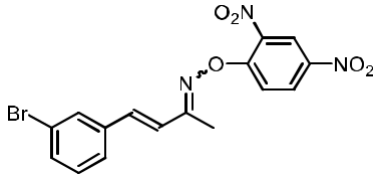
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.7 Hz, 0.50H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.48–8.43 (m, 1.50H), 8.05 (d, *J* = 9.4 Hz, 0.50H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.77 (d, *J* = 16.4 Hz, 0.50H), 7.47–7.34 (m, 2H), 7.30 (d, *J* = 7.7 Hz, 1H), 7.28–7.22 (m, 1.50H), 7.17–7.02 (m, 3H), 6.93 (d, *J* = 16.4 Hz, 1H), 2.42 (s, 3H), 2.33 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.13 (d, *J* = 246.8 Hz), 163.09 (d, *J* = 246.8 Hz), 163.08, 159.9, 157.3, 157.1, 140.9, 140.6, 139.6 (d, *J* = 2.4 Hz), 137.61 (d, *J* = 7.7 Hz), 137.36 (d, *J* = 7.5 Hz), 136.9 (d, *J* = 2.3 Hz), 136.1, 135.6, 130.7 (d, *J* = 8.5 Hz), 130.5 (d, *J* = 8.6 Hz), 129.7, 129.5, 124.3, 123.6, 123.4, 122.4, 122.2, 118.0, 117.3, 117.1, 116.5 (d,

J = 21.6 Hz), 114.7 (d, *J* = 21.9 Hz), 113.7, 113.5, 16.5, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}FN_3O_5$ 345.0756; Found 345.0759.



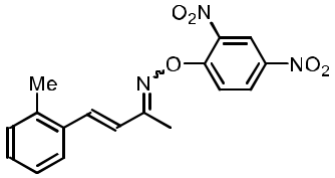
(2E,3E)-4-(3-chlorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1r: an inseparable mixture of *E/Z* isomers (2/1), 1.44 g, 80% yield; yellow solid; m.p. 145–147 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.50H), 8.90 (d, *J* = 2.7 Hz, 1.0H), 8.48–8.43 (m, 1.50H), 8.04 (d, *J* = 9.4 Hz, 0.50H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.76 (d, *J* = 16.5 Hz, 0.50H), 7.56– 7.52 (m, 2H), 7.44–7.31 (m, 4H), 7.10 (d, *J* = 16.5 Hz, 1H), 7.09 (d, *J* = 16.5 Hz, 0.50H), 6.94 (d, *J* = 16.4 Hz, 1H), 2.41 (s, 3H), 2.33 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.0, 159.9, 157.3, 157.1, 140.9, 140.6, 139.4, 137.2, 136.9, 136.6, 136.3, 136.04, 136.00, 135.6, 135.0, 130.4, 130.2, 130.1, 129.6, 129.5, 128.4, 127.1, 125.6, 125.5, 124.4, 122.4, 122.1, 118.1, 117.2, 117.1, 16.5, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}ClN_3O_5$ 361.0465; Found 361.0481.



(2E,3E)-4-(3-bromophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

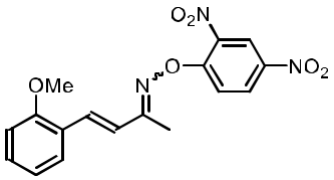
1s: an inseparable mixture of *E/Z* isomers (1/1), 1.52 g, 75% yield; yellow solid; m.p. 148–150 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.8 Hz, 1H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.50–8.37 (m, 2H), 8.04 (d, *J* = 9.4 Hz, 1H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.76 (d, *J* = 16.4 Hz, 1H), 7.70–7.66 (m, 2H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 7.9 Hz, 1H), 7.44 (d, *J* = 7.9 Hz, 1H), 7.31 (t, *J* = 7.9 Hz, 1H), 7.28 (t, *J* = 7.9 Hz, 1H), 7.09 (d, *J* = 16.4 Hz, 1H), 7.08 (d, *J* = 16.4 Hz, 1H), 6.93 (d, *J* = 16.4 Hz, 1H), 2.41 (s, 3H), 2.33 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 163.0, 159.9, 157.3, 157.1, 140.9, 140.7, 139.3, 137.4, 137.2, 136.5, 136.1, 135.7, 133.1, 132.4, 131.5, 130.6, 130.5, 130.0, 129.6, 129.4, 126.0, 125.8, 124.4, 123.2, 123.1, 122.4, 122.1, 118.1, 117.2, 117.1, 16.5, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}BrN_3O_5$ 404.9960; Found 404.9967.



(2E,3E)-4-(o-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

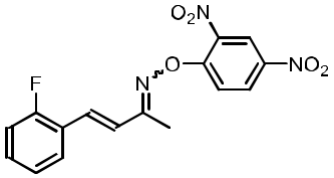
1t: an inseparable mixture of *E/Z* isomers (1.8/1), 1.44 g, 80% yield; yellow solid; m.p. 138–140 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.8 Hz, 0.55H), 8.91 (d, *J* = 2.8 Hz, 1H), 8.47–8.42 (m, 1.55H), 8.05 (d, *J* = 9.5 Hz, 0.55H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.74–7.71 (m, 0.55H), 7.68 (d, *J* = 16.3 Hz, 0.55H), 7.59 (d, *J* = 7.1 Hz, 1H), 7.43 (d, *J* = 16.5 Hz, 1.55H), 7.35–7.14 (m, 4.65H), 6.85 (d, *J* = 16.3 Hz, 1H), 2.46 (s, 1.65H), 2.45 (s, 3H), 2.44 (s, 3H), 2.35 (s, 1.65H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.5, 160.4, 157.4, 157.2, 140.8, 140.5, 138.6, 137.2, 136.6, 136.1, 135.9, 135.7, 134.4, 134.0, 130.9, 130.8, 130.1, 129.6, 129.44, 129.40, 126.8, 126.6, 126.5, 125.9, 124.1, 122.3,

122.1, 117.8, 117.3, 117.1, 19.8, 19.8, 16.5, 12.3. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_5$ 341.1006; Found 341.1013.



(2E,3E)-4-(2-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1u: an inseparable mixture of *E/Z* isomers (1.7/1), 1.25 g, 70% yield; yellow solid; m.p. 142–144 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.8 Hz, 0.60H), 8.90 (d, *J* = 2.8 Hz, 1H), 8.46–8.41 (m, 1.60H), 8.05 (d, *J* = 9.4 Hz, 0.60H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.85 (d, *J* = 16.6 Hz, 0.60H), 7.65 (dd, *J* = 7.8, 1.7 Hz, 0.60H), 7.60–7.45 (m, 2.60H), 7.40–7.31 (m, 1.60H), 7.07–6.89 (m, 4.20H), 3.95 (s, 1.80H), 3.92 (s, 3H), 2.43 (s, 3H), 2.34 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 164.1, 161.0, 158.2, 157.6, 157.5, 157.4, 140.7, 140.4, 136.5, 136.0, 135.6, 133.3, 131.5, 130.8, 129.5, 129.4, 128.8, 127.3, 124.3, 123.9, 122.9, 122.3, 122.1, 121.0, 120.9, 117.3, 117.2, 117.1, 111.1, 55.6, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_6$ 357.0955; Found 357.0956.

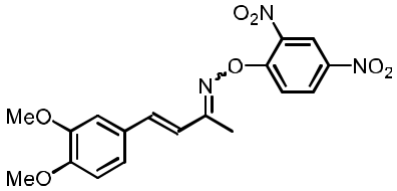


(2E,3E)-4-(2-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1v: an inseparable mixture of *E/Z* isomers (1.7/1), 1.26 g, 73% yield; yellow solid; m.p. 131–133 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.7 Hz, 0.60H), 8.89 (d, *J* = 2.7 Hz, 1H), 8.46–8.42 (m, 1.60H), 8.04 (d, *J* = 9.4 Hz, 1H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.82 (d, *J* = 16.6 Hz, 0.60H), 7.74 (td, *J* = 7.7, 1.5 Hz, 0.60H), 7.59 (td, *J* = 7.7, 1.5 Hz, 1H), 7.41–7.30 (m, 3H), 7.23 (t, *J* = 7.6 Hz, 0.60H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.15–7.08 (m, 1.60H), 7.01 (d, *J* = 16.6 Hz, 1H), 2.42 (s, 3H), 2.35 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.4, 161.2 (d, *J* = 252.9 Hz), 160.8 (d, *J* = 252.0 Hz), 160.3, 157.3, 157.1, 140.9, 140.6, 136.1, 135.6, 132.8 (d, *J* = 4.6 Hz), 131.8 (d, *J* = 8.6 Hz), 131.0 (d, *J* = 8.6 Hz), 130.5 (d, *J* = 3.7 Hz), 129.6, 129.4, 127.70 (d, *J* = 2.8 Hz), 125.11 (d, *J* = 5.3 Hz), 124.8 (d, *J* = 3.7 Hz), 124.5 (d, *J* = 3.7 Hz), 123.4 (d, *J* = 11.5 Hz), 123.1 (d, *J* = 11.4 Hz). 122.3, 122.1, 118.5, 118.4, 117.3, 117.1, 116.2 (d, *J* = 22.1 Hz), 116.1 (d, *J* = 21.9 Hz), 16.4, 12.1.

HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{12}FN_3O_5$ 345.0756; Found 345.0767.

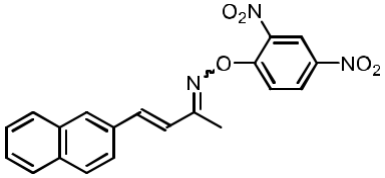


(2E,3E)-4-(3,4-dimethoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1w: an inseparable mixture of *E/Z* isomers (2/1), 1.52 g, 75% yield; yellow solid; m.p. 160–162 °C.

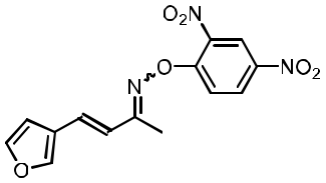
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.7 Hz, 0.50H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.44 (dd, *J* = 9.4, 2.7 Hz, 1.50H), 8.05 (d, *J* = 9.4 Hz, 0.50H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.70 (d, *J* = 16.3 Hz, 0.50H), 7.26–7.24 (m, 0.50H), 7.18–7.06 (m, 4H), 6.90 (d, *J* = 8.1 Hz, 0.50H), 6.89 (d, *J* = 8.1 Hz, 1H), 6.81 (d, *J* = 16.3 Hz, 1H), 4.00 (s, 1.50H), 3.95 (s, 3H), 3.93 (s, 1.50H), 3.93 (s, 3H), 2.41 (s,

3H), 2.31 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 160.5, 157.5, 157.3, 151.2, 150.6, 149.5, 149.3, 140.7, 140.4, 138.2, 136.0, 135.6, 129.6, 129.4, 128.4, 128.2, 122.7, 122.4, 122.1, 121.6, 120.6, 117.2, 116.9, 114.9, 111.2, 111.0, 109.1, 109.0, 56.0, 55.9, 16.2, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{17}N_3O_7$ 387.1061; Found 387.1060.



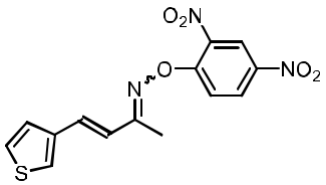
(2E,3E)-4-(naphthalen-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1x: an inseparable mixture of *E/Z* isomers (1.7/1), 1.32 g, 70% yield; yellow solid; m.p. 156–158 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.97 (d, *J* = 2.7 Hz, 0.60H), 8.91 (d, *J* = 2.7 Hz, 1H), 8.52–8.30 (m, 1.60H), 8.06 (d, *J* = 9.3 Hz, 0.60H), 8.05 (d, *J* = 9.3 Hz, 1H), 8.01–7.81 (m, 7.60H), 7.71 (d, *J* = 8.6 Hz, 1H), 7.56–7.49 (m, 3.20H), 7.34 (d, *J* = 16.4 Hz, 1H), 7.36 (d, *J* = 16.4 Hz, 0.60H), 7.06 (d, *J* = 16.3 Hz, 1H), 2.47 (s, 3H), 2.38 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.4, 160.3, 157.5, 157.2, 141.2, 140.8, 140.5, 138.3, 136.0, 135.6, 134.3, 133.9, 133.4, 133.3, 132.8, 132.7, 130.1, 129.6, 129.4, 129.1, 128.8, 128.7, 128.6, 128.4, 127.9, 127.8, 127.3, 127.0, 126.8, 123.3, 123.2, 123.1, 122.4, 122.1, 117.2, 117.1, 116.9, 16.5, 12.3. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{20}H_{15}N_3O_5$ 377.1006; Found 377.1005.



(2E,3E)-N-(2,4-dinitrophenyl)-4-(furan-3-yl)but-3-en-2-imine

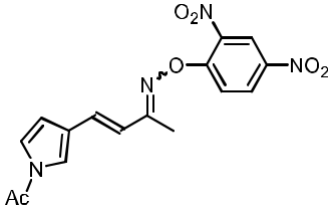
1y: an inseparable mixture of *E/Z* isomers (1.7/1), 0.98 g, 65% yield; yellow solid; m.p. 126–128 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.93 (d, *J* = 2.7 Hz, 0.60H), 8.89 (d, *J* = 2.7 Hz, 1H), 7.46–7.41 (m, 1.60H), 8.02 (d, *J* = 9.4 Hz, 0.60H), 8.01 (d, *J* = 9.4 Hz, 1H), 7.65 (d, *J* = 20.6 Hz, 1.60H), 7.51–7.38 (m, 2.20H), 7.07 (d, *J* = 16.2 Hz, 1.60H), 6.78 (s, 0.60H), 6.63 (d, *J* = 16.1 Hz, 2H), 2.36 (s, 3H), 2.27 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.3, 160.3, 157.4, 157.2, 144.7, 144.4, 144.2, 143.1, 140.7, 140.4, 136.0, 135.6, 131.0, 129.6, 129.4, 128.2, 123.8, 123.5, 122.5, 122.3, 122.1, 117.2, 117.0, 116.8, 107.5, 107.2, 16.2, 12.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_{11}N_3O_5$ 317.0642; Found 317.0628.



(2E,3E)-N-(2,4-dinitrophenyl)-4-(thiophen-3-yl)but-3-en-2-imine

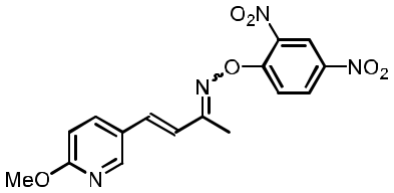
1z: an inseparable mixture of *E/Z* isomers (2.5/1), 1.25 g, 75% yield; yellow solid; m.p. 151–153 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.40H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.46–8.41 (m, 1.40H), 8.04 (d, *J* = 9.3 Hz, 0.40H), 8.03 (d, *J* = 9.4 Hz, 1H), 7.60 (d, *J* = 16.3 Hz, 0.40H), 7.52 (d, *J* = 2.4 Hz, 0.40H), 7.47 (d, *J* = 4.9 Hz, 0.40H), 7.43 (d, *J* = 2.2 Hz, 1H), 7.41–7.31 (m, 2.40H), 7.19 (d, *J* = 16.3 Hz, 1H), 7.17 (d, *J* = 16.3 Hz, 0.40H), 6.77 (d, *J* = 16.3 Hz, 1H), 2.39 (s, 3H), 2.30

(s, 1.20H). **^{13}C NMR** (125 MHz, Acetone) δ 163.9, 161.0, 157.0, 156.8, 141.0, 140.8, 138.9, 138.8, 135.3, 132.7, 129.8, 129.6, 128.4, 127.6, 127.1, 126.5, 125.1, 124.7, 122.3, 122.0, 121.8, 117.2, 117.0, 116.1, 15.5, 11.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_{11}N_3O_4S$ 333.0414; Found 333.0392.



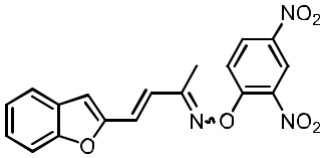
1-(3-((1E,3E)-3-((2,4-dinitrophenyl)imino)but-1-en-1-yl)-1H-pyrrol-1-yl)ethan-1-one

1aa: an inseparable mixture of *E/Z* isomers (1.7/1), 1.20 g, 70% yield; yellow solid; m.p. 169– 171 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.94 (d, *J* = 2.7 Hz, 0.60H), 8.89 (d, *J* = 2.7 Hz, 1H), 8.43 (d, *J* = 9.4 Hz, 1.60H), 8.03 (t, *J* = 8.8 Hz, 1.60H), 7.62–7.41 (m, 2.20H), 7.31 (s, 1.60H), 7.05 (d, *J* = 16.2 Hz, 1.60H), 6.76–6.63 (m, 1.60H), 6.58–6.55 (m, 1H), 2.56 (s, 1.80H), 2.56 (s, 3H), 2.37 (s, 3H), 2.28 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 167.2, 163.4, 160.3, 157.5, 157.3, 140.7, 140.4, 136.0, 135.6, 133.2, 130.4, 129.6, 129.4, 125.8, 125.7, 122.3, 122.2, 122.1, 121.4, 121.2, 121.0, 119.8, 117.2, 117.0, 116.4, 110.8, 110.5, 22.2, 22.2, 16.2, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{14}N_4O_5$ 358.0908; Found 358.0910.



(2E,3E)-4-(6-methoxypyridin-3-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

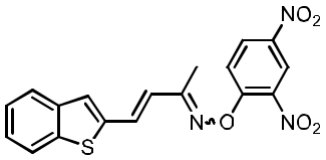
1ab: an inseparable mixture of *E/Z* isomers (2/1), 1.20 g, 67% yield; yellow solid; m.p. 182–184 °C. **^{1}H NMR** (500 MHz, DMSO-*d*$_6$) δ 8.87–8.83 (m, 1.50H), 8.60 (dd, *J* = 9.4, 2.8 Hz, 1H), 8.56 (dd, *J* = 9.4, 2.8 Hz, 0.50H), 8.45–8.43 (dd, *J* = 4.0, 2.4 Hz, 1.50H), 8.14 (dd, *J* = 8.8, 2.5 Hz, 1H), 8.02–7.97 (m, 2H), 7.55–7.39 (m, 2H), 7.00 (d, *J* = 16.5 Hz, 1H), 6.98 (d, *J* = 8.7 Hz, 0.50H), 6.88 (d, *J* = 8.7 Hz, 1H), 3.91 (s, 1.50H), 3.90 (s, 3H), 2.38 (s, 3H), 2.33 (s, 1.50H). **^{13}C NMR** (125 MHz, DMSO-*d*$_6$) δ 165.1, 164.2, 164.4, 161.3, 156.8, 156.6, 148.7, 148.0, 141.0, 140.7, 138.7, 137.2, 136.9, 136.3, 135.9, 135.8, 130.7, 130.6, 125.6, 125.3, 122.6, 122.4, 121.9, 117.5, 117.4, 115.4, 112.0, 111.5, 54.1, 54.0, 16.6, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{14}N_4O_6$ 358.0908; Found 358.0907.



(2E,3E)-4-(benzofuran-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

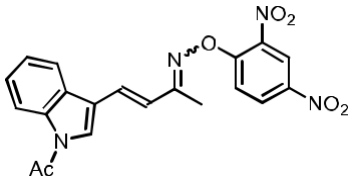
1ac: an inseparable mixture of *E/Z* isomers (1.8/1), 1.28 g, 70% yield; yellow solid; m.p. 195– 197 °C. **^{1}H NMR** (500 MHz, DMSO-*d*$_6$) δ 8.86 (d, *J* = 2.8 Hz, 0.55H), 8.84 (d, *J* = 2.8 Hz, 1H), 8.57 (dd, *J* = 9.4, 2.8 Hz, 1.55H), 8.04 (d, *J* = 9.3 Hz, 1H), 7.99 (d, *J* = 9.3 Hz, 0.55H), 7.73 (d, *J* = 7.7 Hz, 0.55H), 7.69 (d, *J* = 7.7 Hz, 1H), 7.65 – 7.55 (m, 2.10H), 7.48 (d, *J* = 16.2 Hz, 1.55H), 7.45 – 7.36 (m, 1.55H), 7.34 – 7.25 (m, 2.10H), 7.22 (s, 1H), 6.99 (d, *J* = 16.3 Hz, 1H), 2.38 (s, 3H),. 2.34 (s, 1.65H). **^{13}C NMR** (151 MHz, DMSO-*d*$_6$) δ 163.8, 160.7, 156.8, 156.4, 155.4, 155.1, 153.6,

153.3, 141.2, 141.0, 136.4, 136.2, 130.7, 130.6, 129.4, 128.8, 128.6, 127.3, 126.7, 126.6, 124.1, 124.0, 123.7, 122.7, 122.6, 122.40, 122.37, 117.74, 117.71, 116.3, 112.4, 111.8, 111.7, 110.0, 16.7, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{13}N_3O_6$ 367.0799; Found 367.0797.



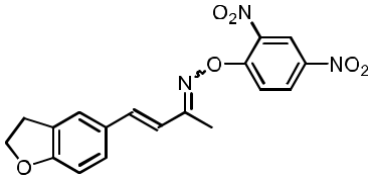
(2E,3E)-4-(benzo[b]thiophen-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1ad: an inseparable mixture of *E/Z* isomers (1.5/1), 1.38 g, 72% yield; yellow solid; m.p. 191– 193 °C. **^{1}H NMR** (500 MHz, DMSO-*d*$_6$) δ 8.87 (d, *J* = 2.8 Hz, 0.65H), 8.85 (d, *J* = 2.8 Hz, 1H), 8.61–8.56 (m, 1.65H), 8.05 (d, *J* = 9.4 Hz, 1H), 8.04 (d, *J* = 7.7 Hz, 0.65H), 8.01 (d, *J* = 9.4 Hz, 0.65H), 7.99–7.95 (m, 1H), 7.93–7.86 (m, 1.65H), 7.84–7.76 (m, 2.30H), 7.72 (s, 1H), 7.48–7.35 (m, 3.95H), 6.75 (d, *J* = 16.1 Hz, 1H), 2.40 (s, 3H), 2.35 (s, 1.95H).**^{13}C NMR** (151 MHz, DMSO-*d*$_6$) δ 163.9, 160.8, 156.7, 156.5, 141.2, 141.00, 140.97, 140.9, 140.0, 139.93, 139.87, 139.4, 136.4, 136.0, 135.9, 133.0, 130.8, 130.6, 130.0, 127.9, 127.0, 126.5, 125.6, 125.5, 125.2, 124.9, 124.2, 123.3, 123.1, 122.6, 122.4, 117.7, 117.5, 117.4, 16.6, 12.4. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{13}N_3O_5S$ 383.0570; Found 383.0573.



1-(3-((1E,3E)-3-((2,4-dinitrophenyl)imino)but-1-en-1-yl)-1H-indol-1-yl)ethan-1-one

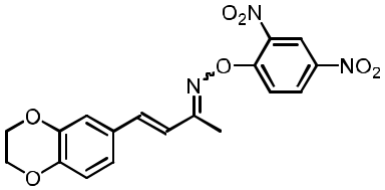
1ae: an inseparable mixture of *E/Z* isomers (5/1), 1.49 g, 60% yield; yellow solid; m.p. 178–180 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.97 (d, *J* = 2.8 Hz, 0.20H), 8.92 (d, *J* = 2.7 Hz, 1H), 8.50 (d, *J* = 7.7 Hz, 1.20H), 8.46 (dd, *J* = 9.4, 2.7 Hz, 1.20H), 8.12 (d, *J* = 7.8 Hz, 0.20H), 8.08 (d, *J* = 9.4 Hz, 0.20H), 8.07 (d, *J* = 9.4 Hz, 1H), 7.90 (d, *J* = 7.4 Hz, 1.20H), 7.75 (s, 0.20H), 7.69 (s, 1H), 7.52–7.38 (m, 2.40H), 7.31 (d, *J* = 16.6 Hz, 1H), 7.30 (d, *J* = 16.6 Hz, 0.20H), 7.09 (d, *J* = 16.6 Hz, 1H), 2.71 (s, 0.60H), 2.70 (s, 3H), 2.46 (s, 3H), 2.36 (s, 0.60H). **^{13}C NMR** (125 MHz, DMSO-*d*$_6$) δ 170.1, 164.6, 156.7, 141.0, 136.3, 136.1, 130.6, 128.5, 128.4, 125.9, 124.5, 122.4, 120.4, 118.3, 117.5, 116.6, 24.3, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{20}H_{16}N_4O_5$ 408.1064; Found 408.1060.



(2E,3E)-4-(2,3-dihydrobenzofuran-5-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

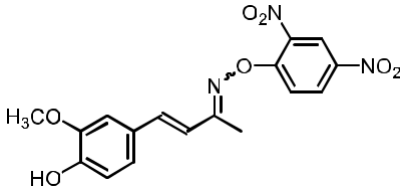
1af: an inseparable mixture of *E/Z* isomers (2/1), 1.48 g, 80% yield; yellow solid; m.p. 157–159 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.7 Hz, 0.50H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.47–8.42 (m, 1.50H), 8.06 (d, *J* = 9.3 Hz, 0.50H), 8.06 (d, *J* = 9.3 Hz, 1H), 7.60 (d, *J* = 16.3 Hz, 0.50H), 7.53 (s, 0.50H), 7.43 (s, 1H), 7.37–7.32 (m, 0.50H), 7.31–7.27 (m, 1H), 7.11 (d, *J* = 16.3 Hz, 1H), 7.10 (d, *J* = 16.3 Hz, 0.50H), 6.86–6.75 (m, 2.50H), 4.68–4.62 (m, 3H), 3.31–3.23 (m, 3H), 2.40 (s, 3H), 2.30 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.7, 162.4, 161.7, 160.6, 157.6, 157.4, 141.3, 140.6, 140.3, 138.3, 136.0, 135.5, 129.9, 129.6, 129.4, 128.6, 128.4, 128.2, 128.2, 128.0, 124.1,

123.6, 122.3, 122.1, 119.8, 117.2, 117.1, 113.6, 109.8, 109.7, 72.0 71.8, 29.4, 16.4, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{15}N_3O_6$ 369.0955; Found 369.0959.



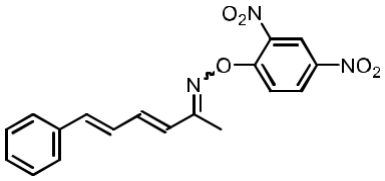
(2E,3E)-4-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1ag: an inseparable mixture of *E/Z* isomers (2/1), 1.64 g, 85% yield; yellow solid; m.p. 173–175 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.7 Hz, 0.50H), 8.90 (d, *J* = 2.6 Hz, 1H), 8.47–8.42 (m, 1.50H), 8.04 (d, *J* = 9.3 Hz, 0.50H), 8.03 (d, *J* = 9.3 Hz, 1H), 7.60 (d, *J* = 16.3 Hz, 0.50H), 7.18–7.10 (m, 1H), 7.08–7.00 (m, 3.50H), 6.90 (d, *J* = 8.3 Hz, 0.50H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.77 (d, *J* = 16.4 Hz, 1H), 4.32–4.26 (m, 6H), 2.39 (s, 3H), 2.29 (s, 1.50H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.5, 160.4, 157.6, 157.3, 145.7, 145.1, 143.8, 140.7, 140.4, 137.8, 136.0, 129.6, 129.4, 129.1, 128.8, 122.3, 122.1, 121.7, 121.2, 121.1, 117.9, 117.8, 117.3, 117.1, 116.9, 115.9, 115.1, 64.6, 64.6, 64.3, 64.2, 16.4, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{15}N_3O_7$ 385.0905; Found 385.0904.



(2E,3E)-4-(4-hydroxy-3-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

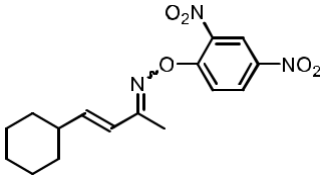
1ah: an inseparable mixture of *E/Z* isomers (1.7/1), 1.21 g, 65% yield; yellow solid; m.p. 159– 161 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.95 (d, *J* = 2.8 Hz, 0.60H), 8.90 (d, *J* = 2.8 Hz, 1H), 8.44 (dd, *J* = 9.4, 2.8 Hz, 1.60H), 8.05 (d, *J* = 9.4 Hz, 0.60H), 8.04 (d, *J* = 9.4 Hz, 1H), 7.68 (d, *J* = 16.3 Hz, 0.60H), 7.17–7.01 (m, 4.80H), 6.94 (d, *J* = 8.3 Hz, 1.60H), 6.79 (d, *J* = 16.3 Hz, 1H), 5.90 (s, 0.60H), 5.83 (s, 1H), 4.02 (s, 1.80H), 3.96 (s, 3H), 2.41 (s, 3H), 2.31 (s, 1.80H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 160.6, 157.5, 157.3, 148.0, 147.3, 147.1, 146.9, 140.9, 140.7, 140.4, 138.3, 136.0, 135.6, 129.6, 129.4, 128.0, 127.8, 123.6, 122.4, 122.1, 120.4, 117.2, 116.9, 114.8, 114.7, 114.6, 108.6, 108.3, 56.0, 16.2, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_7$ 373.0905; Found 373.0905.



(2E,3E,5E)-6-phenylhexa-3,5-dien-2-one O-(2,4-dinitrophenyl) oxime

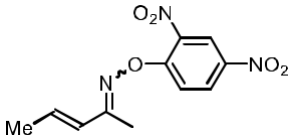
1ai: an inseparable mixture of *E/Z* isomers (2/1), 1.32 g, 75% yield; yellow solid; m.p. 144–146 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.92 (d, *J* = 2.7 Hz, 0.50H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.46–8.43 (m, 1.50H), 8.02 (t, *J* = 9.3 Hz, 1.50H), 7.52–7.45 (m, 3H), 7.40–7.35 (m, 3H), 7.35–7.28 (m, 1.50H), 7.20 (d, *J* = 15.3 Hz, 0.50H), 7.08–6.88 (m, 3.50H), 6.84 (d, *J* = 15.0 Hz, 1H), 6.50 (d, *J* = 14.8 Hz, 1H), 2.36 (d, *J* = 1.2 Hz, 3H), 2.27 (d, *J* = 1.1 Hz, 1.50H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 163.4, 160.2, 157.4, 157.2, 141.4, 140.7, 140.5, 140.1, 138.6, 137.9, 136.2, 136.1, 136.0, 135.8, 129.5, 129.4, 129.2, 128.9, 128.9, 128.8, 127.8, 127.5, 127.2, 127.0, 126.2, 122.2, 122.1, 120.0, 117.3,

117.2, 16.5, 12.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{15}N_3O_5$ 353.1006; Found 353.1007.



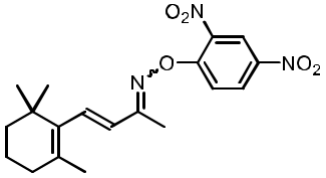
(2E,3E)-4-cyclohexylbut-3-en-2-one O-(2,4-dinitrophenyl) oxime

1aj: an inseparable mixture of *E/Z* isomers (5/1), 1.33 g, 80% yield; yellow solid; m.p. 113–115 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.89 (d, *J* = 2.7 Hz, 0.20H), 8.86 (d, *J* = 2.7 Hz, 1H), 8.40 (dd, *J* = 9.4, 2.7 Hz, 1.20H), 7.98 (d, *J* = 9.4 Hz, 0.20H), 7.96 (d, *J* = 9.4 Hz, 1H), 6.98 (dd, *J* = 16.1, 1.2 Hz, 0.20H), 6.39 (dd, *J* = 16.1, 6.8 Hz, 0.20H), 6.35 (dd, *J* = 16.1, 6.8 Hz, 1H), 6.20 (dd, *J* = 16.1, 1.1 Hz, 1H), 2.25 (s, 3H), 2.22–2.10 (m, 1.60H), 1.86–1.74 (m, 5H), 1.72–1.62 (m, 1.20H), 1.37–1.26 (m, 2.40H), 1.25–1.09 (m, 3.60H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 163.6, 160.6, 157.5, 157.4, 151.2, 147.6, 140.6, 140.4, 136.0, 135.6, 129.5, 129.4, 122.6, 122.2, 122.0, 117.4, 117.2, 117.1, 41.5, 41.2, 32.3, 32.0, 26.0, 25.9, 25.8, 25.8, 16.4, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{19}N_3O_5$ 333.1319; Found 333.1320.



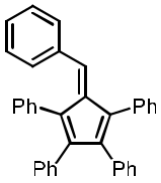
(2E,3E)-pent-3-en-2-one O-(2,4-dinitrophenyl) oxime

1ak: an inseparable mixture of *E/Z* isomers (2/1), 0.93 g, 70% yield; yellow solid; m.p. 80–82 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.91–8.87 (m, 1.50H), 8.44–8.39 (m, 1.50H), 7.99 (d, *J* = 9.4 Hz, 0.50H), 7.97 (d, *J* = 9.4 Hz, 1H), 7.03 (dq, *J* = 15.9, 1.7 Hz, 0.50H), 6.53–6.40 (m, 1.50H), 6.27 (dq, *J* = 15.9, 1.5 Hz, 1H), 2.27 (s, 3H), 2.17 (s, 1.50H), 2.00 (dd, *J* = 6.8, 1.8 Hz, 1.50H), 1.95 (dd, *J* = 6.7, 1.7 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 163.2, 160.2, 157.5, 157.4, 141.2, 140.6, 140.4, 137.2, 136.0, 135.8, 129.4, 129.4, 126.3, 122.1, 122.0, 121.3, 117.2, 117.1, 19.2, 18.8, 16.5, 12.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{11}H_{11}N_3O_5$ 265.0693; Found 265.0702.



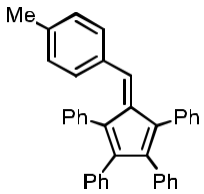
(2E,3E)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime

1al: an inseparable mixture of *E/Z* isomers (1.7/1), 1.31 g, 70% yield; yellow solid; m.p. 76–78 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.91 (d, *J* = 2.7 Hz, 0.60H), 8.90 (d, *J* = 2.7 Hz, 1H), 8.46–8.42 (m, 1.60H), 8.04 (d, *J* = 9.4 Hz, 0.60H), 8.01 (d, *J* = 9.4 Hz, 1H), 7.10 (d, *J* = 16.6 Hz, 0.60H), 6.87 (dd, *J* = 26.3, 16.5 Hz, 1.60H), 6.25 (d, *J* = 16.5 Hz, 1H), 2.34 (s, 3H), 2.24 (s, 1.80H), 2.12 (t, *J* = 6.3 Hz, 1.20H), 2.07 (t, *J* = 6.4 Hz, 2H), 1.85 (d, *J* = 0.9 Hz, 1.80H), 1.76 (d, *J* = 1.1 Hz, 3H), 1.67–1.63 (m, 3.20H), 1.57 (s, 1.80H), 1.53–1.43 (m, 3.20H), 1.12 (s, 3.60H), 1.07 (s, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 163.6, 160.7, 157.6, 157.4, 140.7, 140.6, 140.3, 138.0, 136.7, 136.5, 136.4, 136.0, 135.7, 133.1, 129.5, 129.4, 126.8, 122.2, 122.1, 120.7, 117.3, 117.1, 39.8, 39.5, 34.2, 34.1, 33.7, 33.2, 28.9, 28.8, 21.8, 21.7, 19.0, 18.9, 16.3, 12.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{23}N_3O_5$ 373.1632; Found 373.1637.



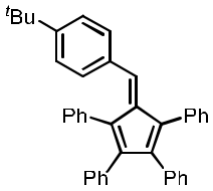
(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

3a: 90% yield (41.2 mg); reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.33–7.25 (m, 5H), 7.10–6.95 (m, 9H), 6.94–6.79 (m, 11H).



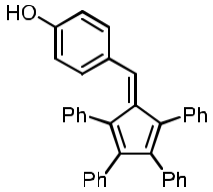
(5-(4-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

3b: 65% yield (30.7 mg); reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.33–7.23 (m, 5H), 7.10–7.00 (m, 6H), 6.96–6.93 (m, 1H), 6.91–6.79 (m, 10H), 6.71 (d, J = 7.9 Hz, 2H), 2.20 (s, 3H).



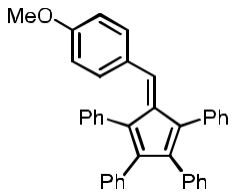
(5-(4-(tert-butyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3c: 46.3 mg, 90% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 202–204 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.47 (s, 1H), 7.34–7.24 (m, 5H), 7.10-7.01 (m, 6H), 6.94–6.78 (m, 13H), 1.22 (s, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.3, 147.0, 145.5, 143.1, 142.9, 138.3, 137.5, 136.9, 136.9, 136.9, 134.3, 133.2, 132.8, 132.4, 132.0, 131.8, 131.6, 129.2, 128.7, 128.6, 128.6, 127.9, 127.7, 127.7, 126.7, 125.4 , 35.9, 32.5. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{34}$ 514.2655; Found 514.2652.



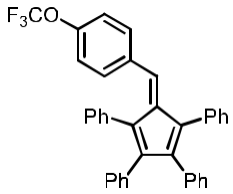
4-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenol

3d: 19.9 mg, 42% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 127–129 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35 (s, 1H), 7.32–7.22 (m, 5H), 7.10–6.99 (m, 6H), 6.98–6.77 (m, 11H), 6.35 (d, J = 8.6 Hz, 2H), 4.84 (br, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.6, 145.6, 142.9, 141.3, 141.2, 137.1, 136.4, 135.6, 135.5, 132.8, 131.8, 131.0, 130.9, 130.6, 130.4, 128.5, 127.76, 127.45, 127.3, 127.2, 126.4, 126.2, 126.1, 125.4, 114.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{26}O$ 474.1978; Found 474.1985.



(5-(4-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

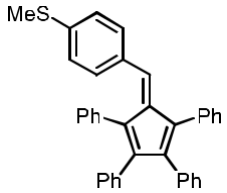
3e: 48% yield (23.4 mg); reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 7.33-7.23 (m, 5H), 7.12–7.01 (m, 6H), 6.98–6.88 (m, 7H), 6.84 (dd, *J* = 17.2, 7.3 Hz, 4H), 6.44 (d, *J* = 8.5 Hz, 2H), 3.70 (s, 3H).



5-(4-(trifluoromethoxy)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

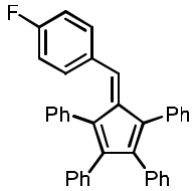
3f: 49.3 mg, 91% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 165–167 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.34–7.23 (m, 5H), 7.13–6.99 (m, 6H), 6.97–6.92 (m, 3H), 6.88–6.83 (m, 4H), 6.83–6.77 (m, 4H), 6.72 (d, *J* = 8.3 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 149.8, 147.7, 147.0, 143.6, 140.4, 137.8, 137.3, 136.6, 136.5, 136.5, 136.0, 133.0, 132.9, 132.5, 132.3, 131.9, 131.7, 129.3, 128.8, 128.6, 128.4 (q, *J* = 286.2 Hz),

128.1, 127.9, 127.8, 127.1, 121.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{25}F_3O$ 542.1852; Found 542.1859.



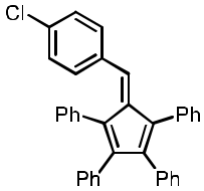
methyl(4-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)sulfane[4]

3g: 75% yield (37.8 mg); reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.37 (d, *J* = 2.9 Hz, 1H), 7.33–7.23 (m, 5H), 7.11–7.02 (m, 6H), 6.99–6.95 (m, 1H), 6.92 (t, *J* = 7.4 Hz, 2H), 6.89–6.85 (m, 6H), 6.85–6.81 (m, 2H), 6.78–6.75 (m, 2H), 2.39 (s, 3H).



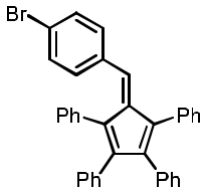
(5-(4-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

3h: 50% yield (23.8 mg); reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.34–7.22 (m, 4H), 7.10–7.01 (m, 6H), 6.99–6.79 (m, 12H), 6.58 (t, *J* = 8.7 Hz, 2H).



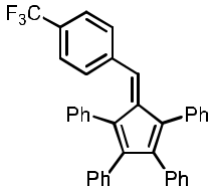
(5-(4-chlorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[5]

3i: 46.5 mg, 94% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35 (s, 1H), 7.34–7.25 (m, 5H), 7.11–7.03 (m, 6H), 7.00 (t, J = 7.8 Hz, 1H), 6.92 (t, J = 7.5 Hz, 2H), 6.89–6.81 (m, 10H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.3, 145.2, 142.1, 139.4, 136.6, 136.1, 135.2, 135.2, 134.2, 133.8, 131.7, 131.6, 131.0, 130.9, 130.5, 130.3, 127.9, 127.5, 127.4, 127.2, 126.6, 126.5, 126.4, 125.7. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}Cl$ 494.1796; Found 494.1802.



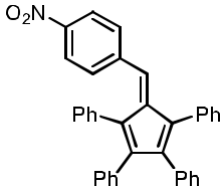
(5-(4-bromobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[6]

3j: 26.8 mg, 50% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35–7.21 (m, 7H), 7.12–6.96 (m, 8H), 6.90 (t, J = 7.6 Hz, 2H), 6.87–6.77 (m, 8H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.3, 145.2, 142.1, 139.4, 136.5, 136.0, 135.2, 135.2, 134.6, 131.8, 131.6, 130.9, 130.5, 130.3, 130.2, 127.9, 127.5, 127.4, 127.2, 126.6, 126.5, 126.4, 125.7, 122.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}Br$ 536.1134; Found 536.1133.



(5-(4-(trifluoromethyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

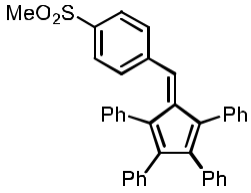
3k: 33.7 mg, 64% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 177–179 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 7.35–7.22 (m, 6H), 7.15–6.99 (m, 9H), 6.92 (t, J = 7.3 Hz, 1H), 6.89–6.75 (m, 8H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.62, 146.49, 142.60, 139.45, 138.59, 136.26, 135.83, 135.06, 135.03, 134.97, 131.59, 131.15, 130.89, 130.47, 130.30, 130.12, 129.4 (q, J = 48.8 Hz), 129.2 (q, J = 129.2 Hz), 127.99, 127.42, 127.39, 127.26, 127.04, 126.77, 126.62, 126.57, 125.87, 124.0 (q, J = 272.2 Hz), 123.81 (q, J = 3.8 Hz). HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{25}F_3$ 526.1903; Found 526.1904.



(5-(4-nitrobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

3l: 45.3 mg, 90% yield ; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1)

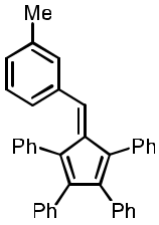
as the eluent. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.7 Hz, 2H), 7.36–7.28 (m, 4H), 7.25 (d, *J* = 6.8 Hz, 2H), 7.12–7.00 (m, 8H), 6.96–6.90 (m, 1H), 6.86–6.82 (m, 4H), 6.82–6.78 (m, 4H).



(5-(4-(methylsulfonyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3m: 49.3 mg, 92% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 209–211 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.3 Hz, 2H), 7.38 (s, 1H), 7.35–7.28 (m, 3H), 7.27–7.24 (m, 2H), 7.14–7.01 (m, 8H), 6.91 (t, *J* = 7.2 Hz, 1H), 6.88–6.76 (m, 8H), 2.94 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 147.4, 147.1, 142.9, 141.7, 138.7, 137.5, 136.4, 135.6, 134.8, 134.8, 134.7, 131.5, 131.0, 130.9, 130.6, 130.3, 130.2, 128.0, 127.43, 127.34,

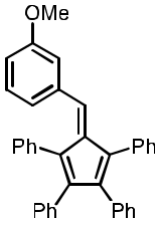
127.3, 126.9, 126.7, 126.7, 125.8, 125.7, 44.3. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{28}O_2S$ 536.1805; Found 536.1813.



(5-(3-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[7]

3n: 29.8 mg, 80% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.34–7.23 (m, 5H), 7.12–7.01 (m, 6H), 6.97–6.79 (m, 12H), 6.76 (s, 1H), 1.98 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.0, 144.1, 141.7, 141.6, 136.9, 136.8, 136.3, 135.5, 135.5, 135.4, 132.1, 131.7, 131.3, 130.9, 130.6, 130.4, 128.7,

127.8, 127.4, 127.3, 127.2, 127.1, 127.1, 126.5, 126.3, 126.2, 125.5, 20.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{28}$ 472.2186; Found 472.2185.

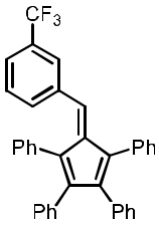


(5-(3-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3o: 43.0 mg, 88% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 198–200 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.34–7.22 (m, 5H), 7.11–7.00 (m, 6H), 6.94–6.78 (m, 10H), 6.62 (d, *J* = 7.5 Hz, 1H), 6.59 (dd, *J* = 8.2, 2.4 Hz, 1H),

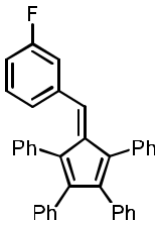
6.47 (s, 1H), 3.48 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.5, 146.3, 144.3, 141.9, 141.3, 137.1, 136.7, 136.2, 135.4, 135.3, 135.2, 131.7, 131.1, 130.9, 130.5, 130.3, 128.2, 127.8, 127.3, 127.3,

127.2, 126.5, 126.4, 126.3, 125.5, 123.0, 115.0, 114.9, 54.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{28}O$ 488.2135; Found 488.2134.



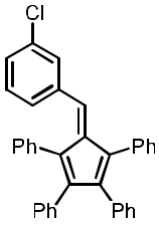
(5-(3-(trifluoromethyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3p: 47.4 mg, 90% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 138–140 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 7.36–7.21 (m, 7H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.12–7.02 (m, 7H), 6.94–6.81 (m, 9H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.8, 146.1, 142.6, 138.6, 136.6, 136.0, 135.9, 135.1, 133.1, 131.6, 131.1, 130.9, 130.5, 130.3, 129.2 (q, *J* = 32.5 Hz), 129.4, 129.1, 128.8 (q, *J* = 211.3 Hz), 128.3 (q, *J* = 28.8 Hz), 128.0, 127.6, 127.5, 127.3, 127.1 (q, *J* = 3.8 Hz), 126.8, 126.6, 126.5, 125.9, 124.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{25}F_3$ 526.1903; Found 526.1903.



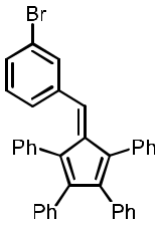
(5-(3-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3q: 34.3 mg, 72% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 144–146 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38–7.24 (m, 6H), 7.12–7.02 (m, 6H), 6.99–6.79 (m, 10H), 6.74 (d, *J* = 7.7 Hz, 1H), 6.70 (t, *J* = 7.7 Hz, 1H), 6.64 (d, *J* = 10.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 161.83 (d, *J* = 245.5 Hz), 146.44, 145.55, 142.27, 139.21, 137.92 (d, *J* = 8.0 Hz), 136.49, 135.99, 135.15, 131.62, 131.12, 130.81, 130.49, 130.30, 128.47 (d, *J* = 8.2 Hz), 127.90, 127.36, 127.21, 126.66, 126.50, 126.44, 126.10, 126.08, 125.80, 117.14, 116.96, 114.55, 114.38. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}F$ 476.1935; Found 476.1935.



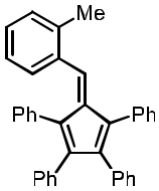
(5-(3-chlorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3r: 39.4 mg, 80% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 178–180 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38–7.24 (m, 6H), 7.12–7.02 (m, 6H), 6.99–6.79 (m, 10H), 6.74 (d, *J* = 7.7 Hz, 1H), 6.70 (t, *J* = 7.7 Hz, 1H), 6.64 (d, *J* = 10.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.5, 145.8, 142.3, 139.0, 137.5, 136.4, 136.0, 135.2, 135.1, 133.3, 131.6, 131.2, 130.8, 130.5, 130.4, 128.3, 128.3, 128.0, 127.7, 127.5, 127.4, 127.3, 126.7, 126.6, 126.5, 126.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}Cl$ 492.1639; Found 492.1638.



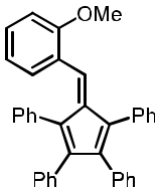
(5-(3-bromobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3s: 47.2 mg, 88% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 145–147 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34–6.24 (m, 5H), 7.16–7.01 (m, 8H), 7.00–6.77 (m, 12H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.5, 145.8, 142.3, 138.8, 137.8, 136.3, 135.9, 135.1, 133.4, 131.6, 131.1, 130.7, 130.5, 130.4, 130.3, 128.6, 128.5, 127.9, 127.5, 127.4, 127.2, 126.7, 126.5, 126.4, 126.0, 121.4. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}Br$ 536.1134; Found 536.1133.



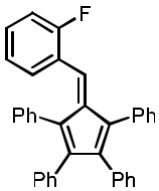
(5-(2-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[4]

3t: 39.2 mg, 83% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.35–7.21 (m, 4H), 7.08–7.00 (m, 6H), 6.96 (d, J = 7.5 Hz, 1H), 6.92–6.75 (m, 11H), 6.70 (d, J = 7.6 Hz, 1H), 6.49 (d, J = 7.5 Hz, 1H), 2.22 (s, 3H).



(5-(2-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene[8]

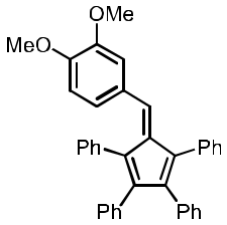
3u: 34.2 mg, 70% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.32–7.22 (m, 5H), 7.13–6.96 (m, 7H), 6.93–6.78 (m, 9H), 6.72 (d, J = 7.5 Hz, 1H), 6.66 (d, J = 8.3 Hz, 1H), 6.28 (t, J = 7.5 Hz, 1H), 3.74 (s, 3H).



(5-(2-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

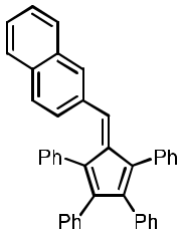
3v: 43.3 mg, 91% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 177–179 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.36–7.27 (m, 5H), 7.15–7.04 (m, 6H), 7.04–6.98 (m, 1H), 6.96–6.77 (m, 11H), 6.53 (t, J = 7.5 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 160.2 (d, J = 248.6 Hz), 146.5, 146.3, 142.3, 136.3, 135.8, 135.2, 135.1, 133.4, 133.4, 132.8, 131.6, 131.4, 130.9, 130.5, 130.4, 129.73 (d, J = 8.1 Hz), 127.9, 127.4, 127.3, 127.2,

126.7, 126.5, 126.4, 125.7, 124.1 (d, J = 14.8 Hz), 122.7 (d, J = 3.3 Hz), 114.4 (d, J = 21.4 Hz). HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{25}F$ 476.1935; Found 476.1937.



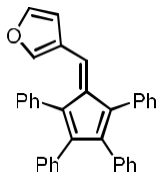
(5-(3,4-dimethoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3w: 35.2 mg, 68% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 87–89 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.34–7.24 (m, 5H), 7.11– 7.02 (m, 6H), 7.00–6.91 (m, 5H), 6.84 (dd, J = 15.8, 7.7 Hz, 4H), 6.71 (d, J = 8.3 Hz, 1H), 6.56 (d, J = 8.3 Hz, 1H), 6.49 (s, 1H), 3.81 (s, 3H), 3.46 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 149.4, 147.8, 146.1, 142.4, 141.9, 141.3, 137.0, 136.6, 135.7, 135.6, 135.5, 131.8, 130.9, 130.6, 130.6, 130.4, 128.7, 127.8, 127.5, 127.32, 127.24, 126.5, 126.3, 126.2, 125.7, 124.7, 114.2, 110.1, 55.9, 55.4. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{38}H_{30}O_2$ 518.2240; Found 518.2237.



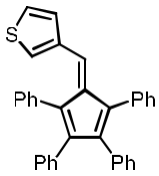
2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)naphthalene

3x: 46.2 mg, 91% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 190–192 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67 (d, J = 8.1 Hz, 1H), 7.61 (s, 1H), 7.52 (s, 1H), 7.47 (d, J = 8.1 Hz, 1H), 7.44–7.39 (m, 2H), 7.37–7.31 (m, 6H), 7.16–7.05 (m, 7H), 7.00–6.84 (m, 6H), 6.82–6.74 (m, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.2, 144.7, 141.8, 141.4, 136.9, 136.5, 135.5, 135.5, 135.4, 133.1, 132.6, 132.5, 131.8, 131.4, 131.3, 130.9, 130.7, 130.4, 128.5, 127.9, 127.8, 127.5, 127.4, 127.3, 127.2, 126.6, 126.6, 126.5, 126.4, 126.3, 125.8, 125.7. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{28}$ 508.2186; Found 508.2176.



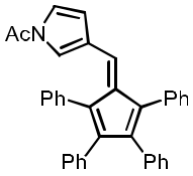
3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)furan

3y: 26.0 mg, 58% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 187–189 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34–7.21 (m, 5H), 7.17–7.12 (m, 5H), 7.11–6.97 (m, 8H), 6.86–6.82 (m, 5H), 5.54 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.7, 147.0, 144.4, 143.8, 142.4, 138.7, 138.0, 137.0, 136.9, 136.9, 133.1, 132.4, 132.1, 132.1, 131.9, 131.7, 129.5, 129.2, 128.7, 128.6, 127.9, 127.8, 127.7, 127.6, 123.5, 113.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{34}H_{24}O$ 448.1822; Found 448.1816.



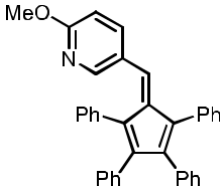
3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)thiophene

3z: 27.8 mg, 60% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 155–157 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34–7.21 (m, 6H), 7.07–6.96 (m, 11H), 6.86 (dd, *J* = 5.0, 3.0 Hz, 1H), 6.85–6.76 (m, 5H), 6.54 (d, *J* = 5.0 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.2, 144.9, 142.8, 138.4, 137.9, 136.9, 136.8, 136.8, 136.0, 133.1, 132.3, 132.0, 131.7, 131.1, 130.1, 129.2, 129.1, 128.7, 128.6, 128.0, 127.9, 127.7, 127.6, 127.3, 125.3. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{34}H_{24}S$ 464.1593; Found 464.1593.



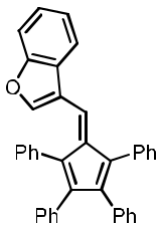
1-(3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-1H-pyrrol-1-yl)ethan-1-one

3aa: 29.8 mg, 61% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 99–100 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.34–7.27 (m, 4H), 7.25–7.22 (m, 2H), 7.14 (s, 5H), 7.10 (s, 1H), 7.09–6.99 (m, 8H), 6.87–6.80 (m, 4H), 2.17 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 167.3, 145.8, 142.5, 140.8, 137.7, 136.7, 135.6, 135.5, 135.4, 133.3, 131.7, 131.1, 130.5, 130.4, 130.3, 128.0, 127.8, 127.3, 127.2, 126.5, 126.3, 126.2, 126.1, 124.0, 123.6, 118.8, 115.7, 22.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{27}NO$ 489.2087; Found 489.2090.



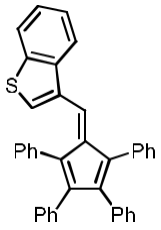
2-methoxy-5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)pyridine

3ab: 19.6 mg, 80% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 166–168 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 2.2 Hz, 1H), 7.36–7.21 (m, 8H), 7.10–7.00 (m, 7H), 6.99–6.92 (m, 3H), 6.91–6.86 (m, 2H), 6.86–6.82 (m, 2H), 6.82–6.77 (m, 2H), 6.20 (d, *J* = 8.6 Hz, 1H), 3.82 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 164.73, 150.45, 147.57, 146.13, 143.22, 141.31, 138.56, 137.98, 137.39, 136.69, 136.66, 133.08, 132.45, 132.22, 131.92, 131.73, 129.27, 129.09, 128.74, 128.59, 128.01, 127.83, 127.75, 127.23, 126.52, 110.51, 54.94. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{27}NO$ 489.2087; Found 489.2086.



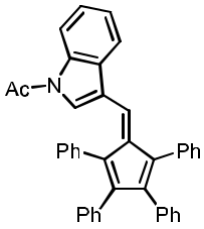
2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)benzofuran[4]

3ac: 35.9 mg, 72% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36–7.26 (m, 6H), 7.24–7.21 (m, 2H), 7.20–7.15 (m, 4H), 7.12–7.00 (m, 8H), 6.95 (d, *J* = 8.2 Hz, 1H), 6.89 (dd, *J* = 7.9, 1.4 Hz, 2H), 6.86 (dd, *J* = 7.9, 1.3 Hz, 2H), 6.09 (s, 1H).



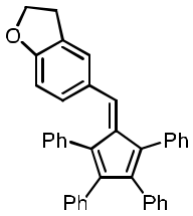
2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)benzo[b]thiophene

3ad: 29.3 mg, 57% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 201–203 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.9 Hz, 1H), 7.35–7.25 (m, 6H), 7.23–7.16 (m, 3H), 7.15–7.11 (m, 2H), 7.10–6.97 (m, 9H), 6.85 (d, *J* = 7.1 Hz, 4H), 6.56 (s, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.0, 146.3, 143.4, 143.2, 140.9, 139.7, 138.4, 137.8, 136.7, 136.6, 136.6, 133.7, 133.1, 132.4, 132.2, 131.9, 131.7, 131.6, 129.3, 128.7, 128.6, 128.1, 128.0, 127.9, 127.8, 127.8, 126.5, 125.6, 125.6, 123.2. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{28}H_{26}S$ 514.1750; Found 514.1744.



1-(2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-1H-indol-1-yl)ethan-1-one

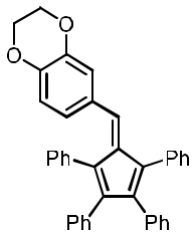
3ae: 30.7 mg, 57% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 196–198 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.35 (d, *J* = 8.2 Hz, 1H), 7.44 (d, *J* = 7.7 Hz, 1H), 7.40 (s, 1H), 7.36–7.27 (m, 7H), 7.16–7.00 (m, 11H), 6.86 (dd, *J* = 7.9, 1.3 Hz, 2H), 6.84–6.79 (m, 2H), 6.38 (d, *J* = 1.3 Hz, 1H), 2.05 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.6, 146.9, 143.7, 141.3, 137.6, 136.7, 135.3, 135.3, 135.2, 135.0, 134.9, 131.7, 131.1, 130.6, 130.3, 130.1, 129.6, 128.9, 128.1, 127.9, 127.4, 127.2, 126.7, 126.5, 126.3, 126.3, 125.6, 124.1, 118.6, 116.9, 116.7, 23.5. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{29}NO$ 539.2244; Found 539.2246.



5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-2,3-dihydrobenzofuran

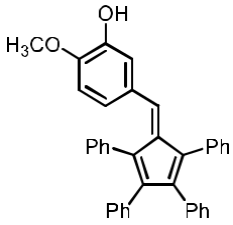
3af: 30.1 mg, 62% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 192–194 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.32–7.21 (m, 5H), 7.10–7.01 (m, 6H), 6.98–6.90 (m, 5H), 6.89–6.77 (m, 5H), 6.73 (s, 1H), 6.45 (d, *J* = 8.3 Hz, 1H), 4.43 (t, *J* = 8.6 Hz, 2H), 2.79 (t, *J* = 8.6 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 160.5, 145.3, 142.2, 142.1, 140.8, 137.4, 136.4, 135.7, 135.6, 131.8, 131.6, 131.0, 130.9, 130.7, 130.4, 128.8, 128.2,

127.7, 127.4, 127.3, 127.1, 126.4, 126.2, 126.2, 126.1, 125.3, 108.2, 71.6, 28.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{38}H_{28}O$ 500.2135; Found 500.2135.



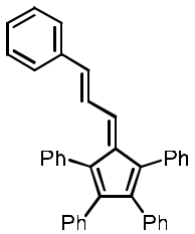
6-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-2,3-dihydrobenzo[b][1,4]dioxin e

3ag: 34.6 mg, 67% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 228–230 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.35–7.24 (m, 4H), 7.13–7.03 (m, 6H), 7.03–6.80 (m, 9H), 6.53–6.49 (m, 2H), 6.46–6.43 (m, 2H), 4.26–4.15 (m, 2H), 4.13–4.06 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 145.6, 143.8, 143.4, 142.4, 141.3, 141.2, 137.1, 136.2, 135.5, 135.5, 135.5, 131.7, 131.1, 130.9, 130.6, 130.4, 129.2, 127.8, 127.3, 127.3, 127.1, 126.4, 126.3, 126.2, 125.3, 124.5, 120.1, 116.0, 64.5, 64.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{38}H_{28}O_2$ 516.2084; Found 516.2084.



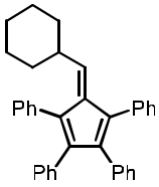
2-methoxy-5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenol

3ah: 32.3 mg, 64% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 86–88 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.35–7.24 (m, 5H), 7.13– 7.02 (m, 6H), 7.01–6.91 (m, 5H), 6.87–6.80 (m, 4H), 6.70 (dd, J = 8.3, 1.5 Hz, 1H), 6.66 (d, J = 8.2 Hz, 1H), 6.46 (d, J = 1.7 Hz, 1H), 5.62 (s, 1H), 3.45 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.2, 146.1, 145.3, 142.1, 142.1, 141.2, 137.1, 136.7, 135.7, 135.5, 135.5, 131.8, 131.0, 130.6, 130.5, 130.4, 128.2, 127.7, 127.5, 127.3, 127.2, 126.4, 126.3, 126.1, 125.5, 125.3, 114.0, 113.5, 55.5. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{37}H_{28}O_2$ 504.2084; Found 504.2064.



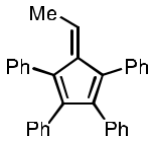
(E)-(5-(3-phenylallylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene [8]

3ai: 40.2 mg, 83% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42–7.27 (m, 8H), 7.26–7.17 (m, 5H), 7.07–7.01 (m, 6H), 7.00–6.90 (m, 5H), 6.89–6.84 (m, 2H), 6.84–6.77 (m, 1H), 6.64 (d, J = 15.2 Hz, 1H).



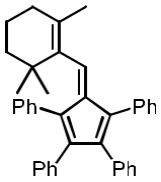
(5-(cyclohexylmethylene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3aj: 34.4 mg, 74% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 206–208 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37–7.22 (m, 7H), 7.18 (d, *J* = 7.7 Hz, 2H), 7.07–6.98 (m, 6H), 6.88 (dd, *J* = 21.4, 6.5 Hz, 4H), 6.27 (d, *J* = 10.9 Hz, 1H), 2.09 (q, *J* = 10.5 Hz, 1H), 1.68–1.45 (m, 6H), 1.08–0.92 (m, 3H), 0.82–0.70 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 151.5, 144.1, 142.8, 140.5, 138.5, 135.7, 135.7, 135.5, 135.1, 132.1, 131.6, 130.6, 130.4, 130.2, 127.7, 127.6, 127.3, 127.1, 126.6, 126.3, 126.1, 126.1, 37.7, 32.6, 25.7, 25.3. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{32}$ 464.2499; Found 464.2502.



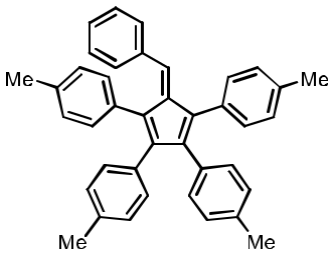
(5-ethylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene [9]

3ak: 34.9 mg, 88% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34–7.16 (m, 10H), 7.08–6.99 (m, 6H), 6.98–6.82 (m, 4H), 6.58–6.51 (m, 1H), 1.70 (d, *J* = 7.8 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.9, 146.1, 142.4, 141.9, 139.6, 137.1, 137.0, 136.9, 136.4, 133.3, 132.9, 132.2, 131.8, 131.6, 129.3, 129.2, 128.7, 128.6, 127.9, 127.8, 127.6, 127.5, 17.8.



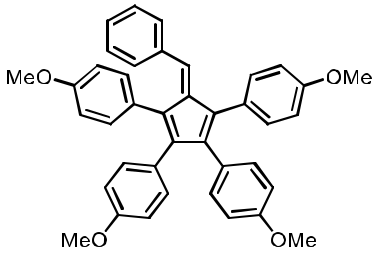
(5-((2,6,6-trimethylcyclohex-1-en-1-yl)methylene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene

3al: 18.1 mg, 36% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 151–153 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.30–7.26 (m, 2H), 7.25–7.18 (m, 3H), 7.16–7.13 (m, 2H), 7.12–7.08 (m, 3H), 7.07–6.96 (m, 6H), 6.89–6.80 (m, 5H), 1.65–1.57 (m, 1H), 1.52–1.44 (m, 1H), 1.38 (s, 3H), 1.36–1.28 (m, 4H), 1.01 (s, 3H), 0.98 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 145.9, 144.9, 142.1, 140.2, 137.0, 136.0, 135.9, 135.9, 135.7, 134.7, 133.6, 132.1, 131.6, 131.5, 130.6, 130.4, 127.6, 127.2, 127.1, 126.6, 126.3, 126.1, 126.0, 125.6, 38.7, 35.0, 31.8, 29.3, 29.3, 23.0, 18.8. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{39}H_{36}$ 504.2812; Found 504.2812.



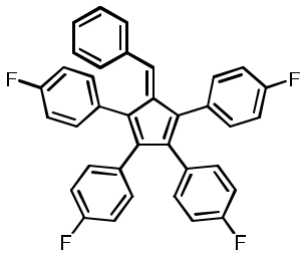
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methylbenzene)

3am: 41.7 mg, 81% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 170–172 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 2.4 Hz, 1H), 7.18–7.10 (m, 4H), 6.99 ((t, *J* = 5 Hz, 1H), 6.94 (d, *J* = 6.9 Hz, 2H), 6.92–6.84 (m, 6H), 6.78–6.70 (m, 6H), 6.66 (d, *J* = 6.4 Hz, 2H), 2.37 (s, 3H), 2.25 (d, *J* = 3.9 Hz, 6H), 2.18 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 145.6, 144.8, 141.5, 140.4, 136.0, 135.9, 135.7, 135.6, 134.7, 133.8, 132.6, 132.5, 131.6, 130.8, 130.5, 130.4, 130.3, 128.6, 128.1, 128.0, 127.9, 127.2, 126.9, 21.3, 21.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{34}$ 514.2655; Found 514.2650.



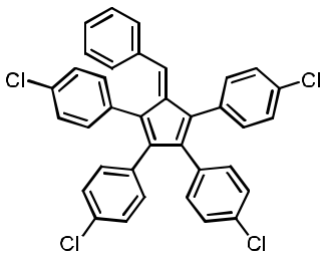
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methoxybenzene) [8]

3an: 46.3 mg, 80% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.18 (d, *J* = 7.7 Hz, 2H), 7.00 (t, *J* = 6.8 Hz, 1H), 6.97–6.89 (m, 4H), 6.86 (d, *J* = 8.3 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 2H), 6.75 (d, *J* = 8.6 Hz, 4H), 6.61 (t, *J* = 7.7 Hz, 4H), 6.43 (d, *J* = 8.0 Hz, 2H), 3.82 (s, 3H), 3.73 (s, 3H), 3.72 (s, 3H), 3.68 (s, 3H).



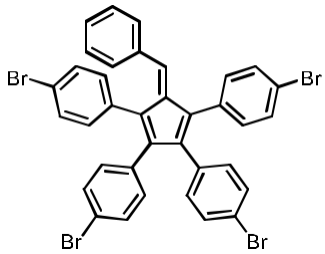
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene)

3ao: 42.4 mg, 80% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 158–160 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.24 (dd, *J* = 8.1, 5.7 Hz, 2H), 7.13–7.07 (m, 1H), 7.06 (t, *J* = 8.7 Hz, 2H), 7.01–6.97 (m, 4H), 6.87–6.77 (m, 10H), 6.62 (t, *J* = 8.7 Hz, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.0 (d, *J* = 255 Hz), 161.6 (d, *J* = 255 Hz), 161.6 (d, *J* = 255 Hz), 161.3 (d, *J* = 256 Hz), 144.92, 144.17, 141.68, 140.70, 135.34, 135.28, 133.05 (d, *J* = 7.8 Hz), 132.35 (d, *J* = 3.3 Hz), 132.25 (d, *J* = 7.8 Hz), 132.07 (d, *J* = 7.9 Hz), 131.84 (d, *J* = 7.8 Hz), 130.92, 130.88, 130.85, 130.83, 130.43, 128.12, 127.27, 115.16, 114.99, 114.79, 114.64 (d, *J* = 4.1 Hz), 114.50 (d, *J* = 2.8 Hz), 114.34. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{22}F_4$ 530.1652; Found 530.1652.



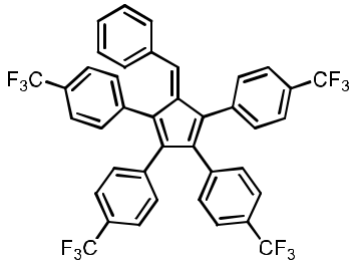
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(chlorobenzene) [8]

3ap: 52.3 mg, 88% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.46–7.42 (m, 1H), 7.34–7.29 (m, 2H), 7.20–7.14 (m, 2H), 7.13–7.05 (m, 5H), 7.01–6.93 (m, 4H), 6.87–6.84 (m, 2H), 6.79–6.68 (m, 6H).



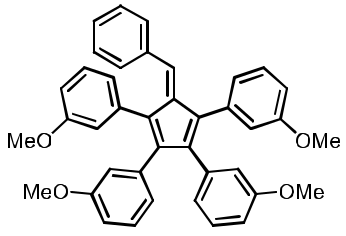
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(bromobenzene)

3aq: 35.4 mg, 46% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 222–224 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.52 (d, J = 8.5 Hz, 1H), 7.48 (d, J = 8.4 Hz, 2H), 7.45 (s, 1H), 7.43–7.39 (m, 1H), 7.28–7.22 (m, 4H), 7.16–7.09 (m, 3H), 7.04 – 7.00 (m, 2H), 7.00–6.93 (m, 3H), 6.75–6.69 (m, 2H), 6.69–6.63 (m, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.5, 143.6, 142.6, 140.4, 135.6, 135.1, 135.0, 133.6, 133.5, 133.0, 132.3, 132.0, 131.8, 131.7, 131.3, 131.0, 130.9, 130.8, 130.7, 130.4, 128.4, 127.4, 122.8, 121.9, 121.3, 121.2, 121.1, 120.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{22}Br_4$ 769.8449; Found 769.8420.



4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis((trifluoromethyl)benzene)

3ar: 25.6 mg, 35% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 88–90 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.60 (d, J = 8.2 Hz, 2H), 7.49 (s, 1H), 7.38–7.32 (m, 6H), 7.12 (d, J = 8.1 Hz, 2H), 7.07–7.03 (m, 1H), 6.95–6.85 (m, 10H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.5, 144.3, 143.4, 140.7, 139.6, 138.1, 137.9, 136.5, 134.8, 134.7, 131.73, 131.69, 130.9, 130.5, 130.3, 129.3 (q, J = 31.5 Hz), 129.14 (q, J = 32.8 Hz), 129.07 (q, J = 32.8 Hz), 128.8, 128.2 (q, J = 32.4 Hz), 127.4, 125.1 (q, J = 4.0 Hz), 124.9 (q, J = 3.9 Hz), 124.7 (q, J = 4.1 Hz), 124.50 (q, J = 272.2 Hz), 124.48 (q, J = 3.8 Hz), 124.2 (q, J = 272.2 Hz), 124.1 (q, J = 272.2 Hz), 124.0 (q, J = 272.2 Hz). HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{22}F_{12}$ 730.1524; Found 730.1543.

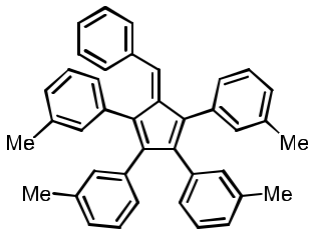


3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methoxybenzene)

3as: 54.4 mg, 94% yield; reddish-brown jelly; purified by using petroleum ether/ethyl acetate (20/1)

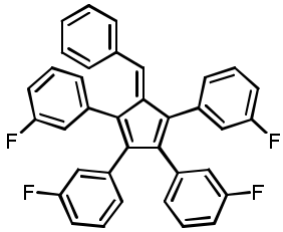
as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.23 (d, *J* = 7.7 Hz, 1H), 7.09–6.92 (m, 7H), 6.90 (d, *J* = 7.5 Hz, 1H), 6.87–6.79 (m, 3H), 6.65 (d, *J* = 8.2 Hz, 2H), 6.52 (d, *J* = 8.0 Hz, 3H), 6.48–6.43 (m, 2H), 6.41 (s, 2H), 3.69 (s, 3H), 3.45 (s, 3H), 3.43 (s, 3H), 3.39 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 159.1, 158.9, 158.6, 158.5, 145.8, 144.0, 141.7, 141.5, 137.9, 136.7, 136.6, 136.5, 136.1, 135.8, 131.2, 130.5, 128.8, 128.4, 128.3, 128.2, 127.9, 127.1, 124.3, 123.6, 123.2,

122.9, 116.9, 115.5, 115.3, 115.0, 113.3, 113.2, 112.7, 55.2, 54.9. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{34}O_4$ 578.2452; Found 578.2453.



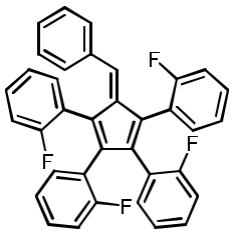
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methylbenzene)

3at: 37.0 mg, 72% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 138–140 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 5.1 Hz, 1H), 7.20 (td, *J* = 7.4, 3.8 Hz, 1H), 7.15–6.86 (m, 12H), 6.84–6.78 (m, 1H), 6.74–6.62 (m, 7H), 2.33 (s, 3H), 2.09 (s, 6H), 1.93 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 145.9, 144.8, 141.8, 140.8, 137.1, 136.6, 136.5, 136.4, 136.3, 136.1, 135.9, 135.5, 135.4, 135.3, 132.4, 132.0, 131.5, 131.3, 131.1, 130.3, 128.8, 127.9, 127.7, 127.6, 127.5, 127.5, 127.2, 127.1, 127.0, 127.0, 126.9, 126.9, 126.1, 21.6, 21.4, 21.1. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{40}H_{34}$ 514.2655; Found 514.2647.



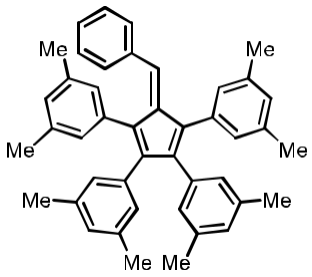
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene)

3au: 37.1 mg, 70% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 150–152 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.47 (s, 1H), 7.34–7.27 (m, 1H), 7.12–6.92 (m, 10H), 6.91–6.82 (m, 3H), 6.69–6.46 (m, 7H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.4 (d, *J* = 247.0 Hz), 162.2 (d, *J* = 244.4 Hz), 162.2 (d, *J* = 245.8 Hz), 162.0 (d, *J* = 245.7 Hz), 144.9, 143.5, 143.0, 140.6, 140.6, 138.3, 138.2, 136.9, 136.9, 136.8, 136.8, 136.8, 135.7, 135.1, 130.9, 130.4, 129.5 (d, *J* = 8.5 Hz), 129.2 (d, *J* = 8.2 Hz), 129.0 (d, *J* = 8.5 Hz), 128.9 (d, *J* = 8.5 Hz), 128.5, 127.3, 127.3 (d, *J* = 2.9 Hz), 126.5 (d, *J* = 2.9 Hz), 126.1 (d, *J* = 2.9 Hz), 125.8 (d, *J* = 2.7 Hz), 118.26 (d, *J* = 21.3 Hz), 117.46 (d, *J* = 21.6 Hz), 117.05 (d, *J* = 21.9 Hz), 116.82 (d, *J* = 21.8 Hz), 114.0 (d, *J* = 20.2 Hz), 113.9 (d, *J* = 20.2 Hz), 113.9 (d, *J* = 21.4 Hz), 112.9 (d, *J* = 21.1 Hz). HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{22}F_4$ 530.1652; Found 530.1652.



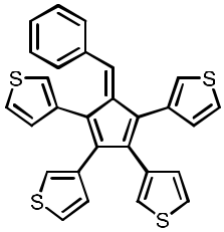
2,2',2'',2'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene)

3av: 44.5 mg, 84% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 185–187 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.33–7.26 (m, 1H), 7.17–6.76 (m, 18H), 6.72 (br, 1H), 6.58 (br, 1H).**^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.0 (d, J = 248.2 Hz), 161.4 (d, J = 247.0 Hz), 161.3 (d, J = 248.2 Hz), 144.45, 144.28, 143.32, 139.81, 136.47, 134.83, 133.74, 132.96, 132.71, 131.46, 130.63 (d, J = 8.1 Hz), 130.44 (d, J = 8.1 Hz), 130.34 (d, J = 8.0 Hz), 129.74 (d, J = 8.1 Hz), 129.48, 128.58, 128.51, 126.07 (d, J = 16.2 Hz), 125.02, 124.75, 124.68, 124.66, 124.62, 124.03 (d, J = 16.1 Hz), 117.11, 116.93, 116.64, 116.46, 116.33, 116.28, 116.10. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{36}H_{22}F_4$ 530.1652; Found 530.1666.



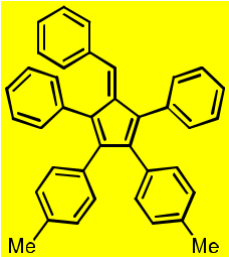
5,5',5'',5'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(1,3-dimethylbenzene)

3aw: 42.8 mg, 75% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 128–130 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 7.01 (d, J = 7.1 Hz, 3H), 6.97–6.90 (m, 5H), 6.71 (s, 2H), 6.57–6.47 (m, 7H), 2.30 (s, 6H), 2.08 (s, 12H), 1.96 (s, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.0, 145.3, 141.8, 140.2, 136.8, 136.4, 136.4, 136.2, 135.9, 135.8, 135.8, 135.6, 135.4, 135.3, 131.0, 130.2, 129.6, 129.0, 128.6, 128.4, 128.0, 127.7, 127.6, 127.3, 126.9, 126.8, 21.4, 21.2, 21.0. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{44}H_{42}$ 570.3281; Found 570.3294.



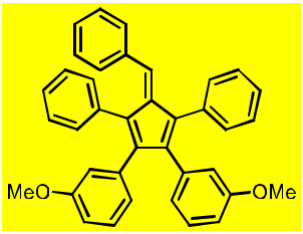
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrathiophene

3ax: 24.1 mg, 50% yield; reddish-brown solid; purified by using petroleum ether/ethyl acetate (20/1) as the eluent; m.p. 194–196 °C. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 (s, 1H), 7.30 (dd, J = 4.8, 3.0 Hz, 1H), 7.12 (d, J = 5.0 Hz, 1H), 7.11–6.97 (m, 7H), 6.92 (d, J = 4.7 Hz, 1H), 6.88 (dd, J = 4.8, 3.0 Hz, 1H), 6.72 (s, 2H), 6.62 (d, J = 5.0 Hz, 1H), 6.60 (d, J = 4.8 Hz, 1H), 6.55 (d, J = 4.8 Hz, 1H), 6.50 (d, J = 4.7 Hz, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.2, 141.6, 140.6, 136.72, 136.70, 135.63, 135.56, 135.5, 135.3, 131.0, 130.3, 130.2, 129.7, 129.4, 129.2, 127.9, 127.3, 126.4, 124.8, 124.5, 124.4, 123.9, 123.8, 123.68, 123.66. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{28}H_{18}S_4$ 482.0286; Found 482.0282.



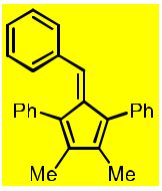
4,4'-(4-benzylidene-3,5-diphenylcyclopenta-2,5-diene-1,2-diyl)bis(methylbenzene)

3ay: 23.3 mg, 48% yield of four inseparable regioisomers (r:r ~ 1:1:1:1); reddish-brown colloid; purified by using petroleum ether/ethyl acetate (50/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45–7.39 (m, 1H), 7.33–7.26 (m, 2H), 7.17–6.98 (m, 6H), 6.97–6.82 (m, 10H), 6.77–6.64 (m, 4H), 2.39–2.15 (m, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.00, 145.96, 145.60, 145.56, 144.60, 144.5, 141.72, 141.68, 141.49, 141.46, 141.1, 140.84, 140.78, 140.5, 136.9, 136.1, 136.0, 135.9, 135.83, 135.80, 135.78, 135.74, 135.72, 135.6, 135.52, 134.8, 133.5, 132.3, 132.24, 132.23, 131.7, 131.5, 131.3, 131.1, 130.94, 130.88, 130.7, 130.6, 130.5, 130.43, 130.41, 130.36, 130.3, 130.21, 130.17, 128.5, 128.0, 127.9, 127.8, 127.7, 127.5, 127.4, 127.30, 127.27, 127.24, 127.18, 127.1, 127.0, 126.9, 126.3, 126.2, 126.1, 125.3, 21.2, 21.0. **HRMS (EI)** m/z: $[M]^+$ Calcd for $C_{38}H_{30}$ 486.2342; Found 486.2342.



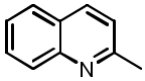
3,3'-(4-benzylidene-3,5-diphenylcyclopenta-2,5-diene-1,2-diyl)bis(methoxybenzene)

3az: 34.7 mg, 67% yield of four inseparable regioisomers (r:r ~ 1:1:1:1); reddish-brown colloid; purified by using petroleum ether/ethyl acetate (10/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.56 (s, 0.5H), 7.49 (s, 0.5H), 7.38 – 7.23 (m, 3H), 7.15 – 6.82 (m, 15H), 6.70 – 6.64 (m, 1H), 6.56 – 6.37 (m, 3H), 3.70 – 3.39 (m, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.9, 158.7, 158.5, 158.42, 158.36, 158.3, 146.05, 146.03, 145.7, 145.6, 144.24, 144.15, 144.0, 143.9, 141.71, 141.69, 141.5, 141.41, 141.40, 141.36, 137.81, 137.76, 136.72, 136.68, 136.6, 136.5, 136.4, 136.3, 136.22, 136.16, 136.0, 135.9, 135.8, 135.5, 135.42, 135.38, 135.3, 135.2, 131.6, 131.22, 131.20, 131.03, 131.00, 130.8, 130.44, 130.41, 130.37, 130.2, 128.7, 128.24, 128.20, 128.1, 128.02 , 127.8, 127.6, 127.33, 127.29, 127.26, 127.2, 127.1, 127.0, 126.53, 126.52, 126.34, 126.28, 125.5, 124.2, 123.5, 123.14, 123.11, 122.8, 116.8, 115.5, 115.2, 114.9, 113.24, 113.21, 113.18, 113.15, 112.5, 55.0, 54.8, 54.7. **HRMS (EI)** m/z: $[M]^+$ Calcd for $C_{38}H_{30}O_2$ 518.2240; Found 518.2256.



(2-benzylidene-4,5-dimethylcyclopenta-3,5-diene-1,3-diyl)dibenzene

3ba: 10.7 mg, 32% yield of three inseparable regioisomers (r:r ~ 2:2:1); reddish-brown colloid; purified by using petroleum ether/ethyl acetate (50/1) as the eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 – 7.30 (m, 9H), 7.18 (s, 0.4H), 7.07 (s, 0.2H), 7.02 – 6.82 (m, 5H), 2.18 – 1.74 (m, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.9, 146.3, 145.0, 144.3, 143.6, 143.41, 142.36, 138.3, 138.2, 137.4, 137.3, 137.24, 137.19, 136.4, 136.3, 135.70, 135.67, 134.5, 134.0, 133.4, 131.0, 130.33, 130.27, 130.24, 130.18, 130.1, 129.6, 129.53, 129.47, 128.03, 127.99, 127.91, 127.86, 127.3, 127.2, 127.1, 127.0, 126.92, 126.86, 126.7, 126.4, 126.3, 125.8, 125.3, 125.2, 15.1, 13.5, 12.2, 12.2, 10.8. **HRMS (EI)** m/z: $[M]^+$ Calcd for $C_{26}H_{22}$ 334.1716; Found 334.1718.



2-methylquinoline[10]

4, 5.0 mg, 35% yield; purified by using petroleum ether/ethyl acetate (20/1) (20/1) as the eluent; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.09–8.00 (m, 2H), 7.78 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.69 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.49 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 1H), 2.76 (s, 3H).

6. References

[1] (a) Shih, J.-L.; Nguyen, T. S.; May, J. A. *Angew. Chem. Int. Ed.* **2015**, *54*, 9931–9935. (b) Hao, H.-Y.; Mao, Y.-J.; Xu, Z.-Y.; Lou, S.-J.; Xu, D.-Q. *Org. Lett.* **2020**, *22*, 2396–2402. (c) Komatsuki, K.; Kozuma, A.; Saito, K.; Yamada, T. *Org. Lett.* **2019**, *21*, 6628–6632. (d) Liu, J., Krajangsri, S., Yang, J.; Li, J.-Q.; Andersson, P. G. *Nat. Catal.* **2018**, *1*, 438–443.

[2] Li, H.-Y.; Wang, M.-L.; Liu, Y.-W.; Li, L.-J.; Xu, H.; Dai, H.-X. *ACS Catal*. **2022**, *12*, 82−88.

[3] (a) Park, K.; Bae, G.; Moon, J.; Choe, J.; Song, K. H.; Lee, S. *J. Org. Chem.* **2010**, *75*, 6244–6251; (b) Lee, J.-H.; Raja, G. C. E.; Son, Y.; Jang, J.; Kim, J.; Lee, S. *Tetrahedron Lett.* **2016**, *57*, 4824–4828.

[4] Peng, S.; Chen, N.; Zhang, H.; He, M.; Li, H.; Lang, M.; Wang, J. *Org. Lett.* **2020**, *22*, 5589−5593.

[5] Taber, D.; Picus, N.; Becker, EI.; Spoerri, PE. *J. Am. Chem. Soc.* **1995**, *77*, 1010−1012.

[6] Alig, I.; Braun, D.; Langendorf, R.; Wirth, H. O.; Voigtb, M.; Wendorff, J. H. *J. Mater. Chem.,* **1998***,8,* 847−851.

[7] Hellwinkel, D.; GöKe, K.; Karle, R. *Synthesis*, **1994**, *9*, 973−978.

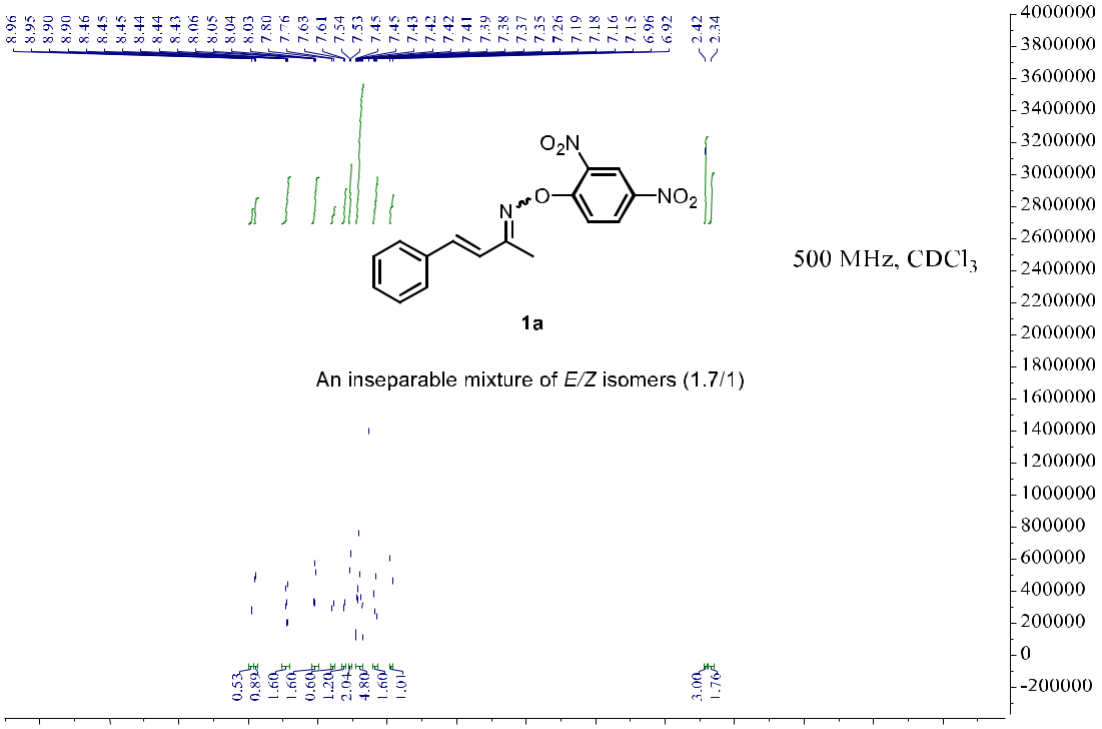
[8] Shibata, K.; Satoh, T.; Miura, M. *Adv. Synth. Catal.* **2007**, *349*, 2317–2325.

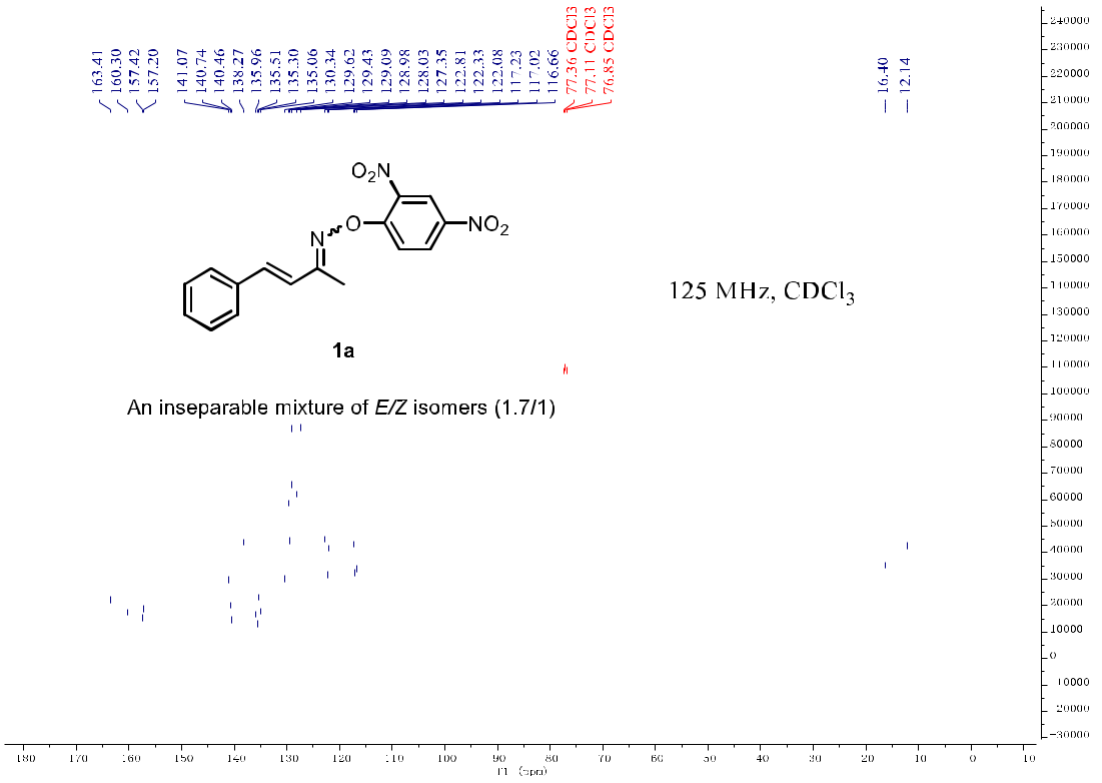
[9] Linder, S. M.; Becker, E. I.; Spoerri, P. E. *J. Am. Chem. Soc.* **1953**, *75*, 5972–5975.

[10] Álvarez-Bercedo, P.; Martin, R. *J. Am. Chem. Soc.* **2010**, *132*, 17352−17353.

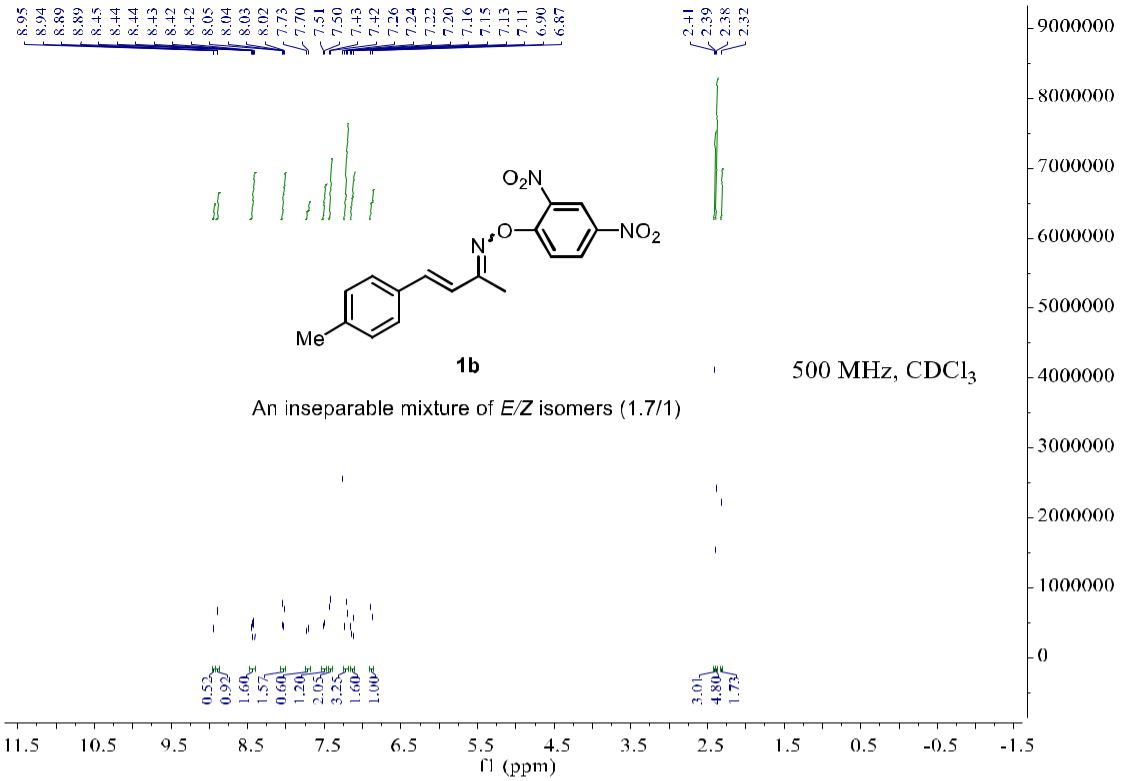
7. NMR Spectra

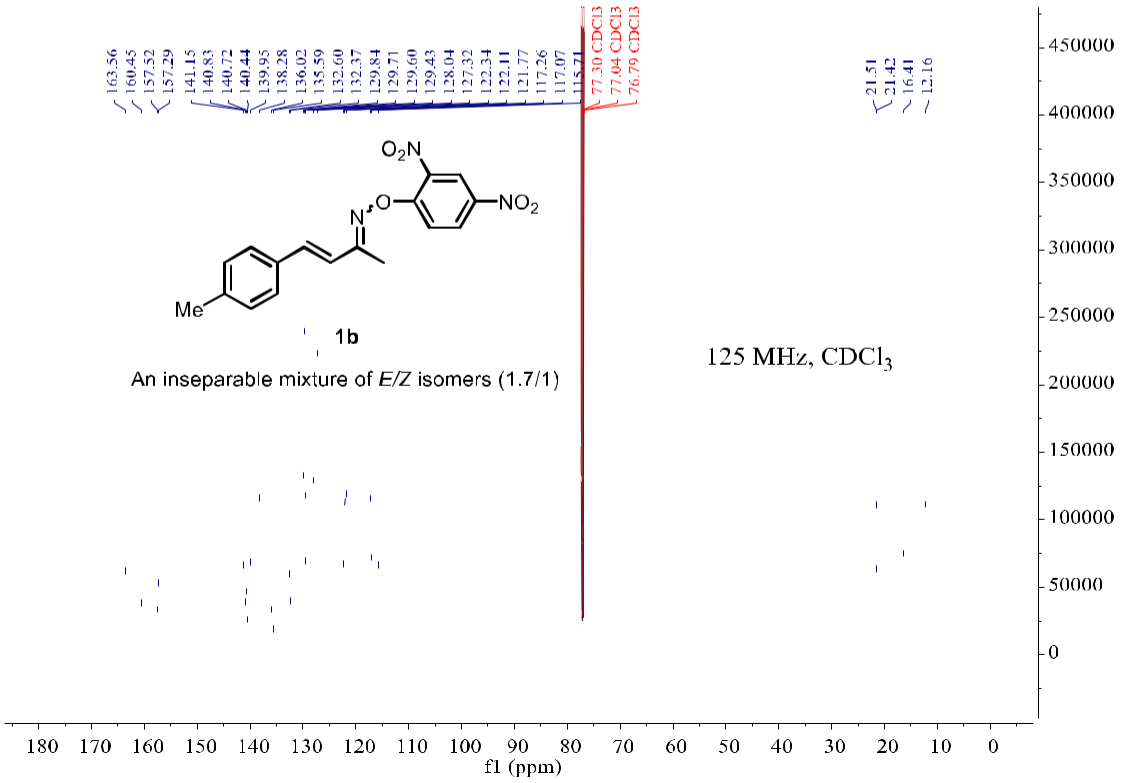
(2E,3E)-4-phenylbut-3-en-2-one O-(2,4-dinitrophenyl) oxime (1a)



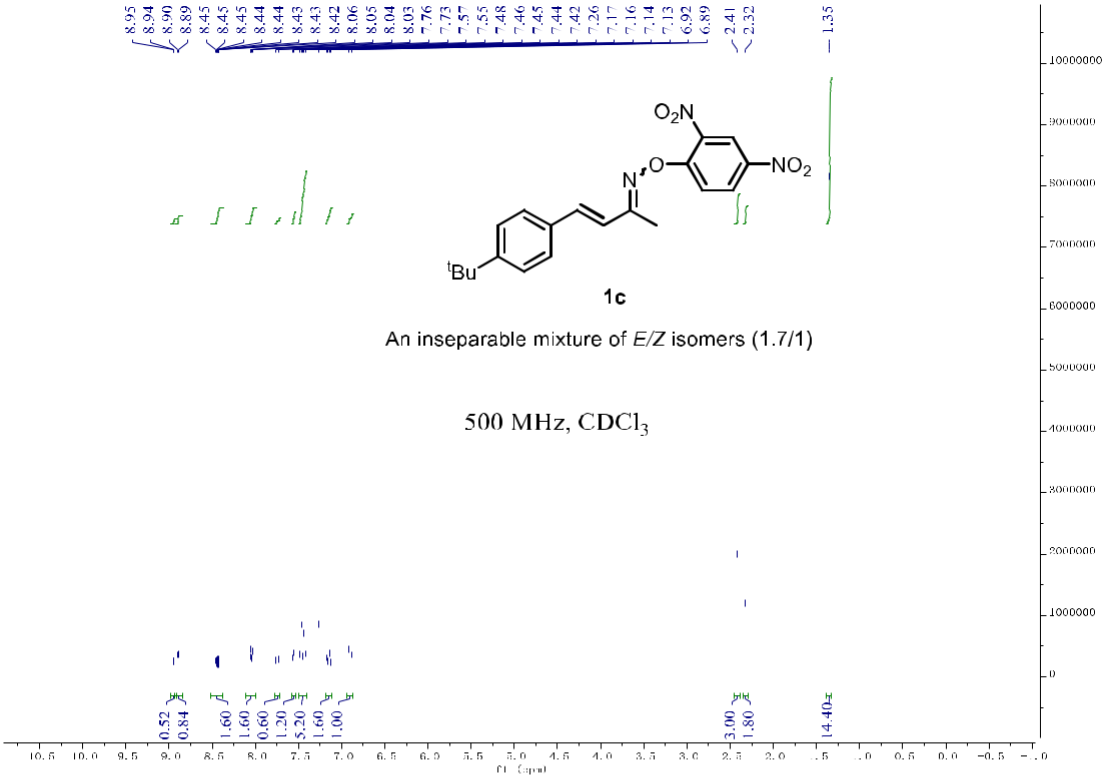


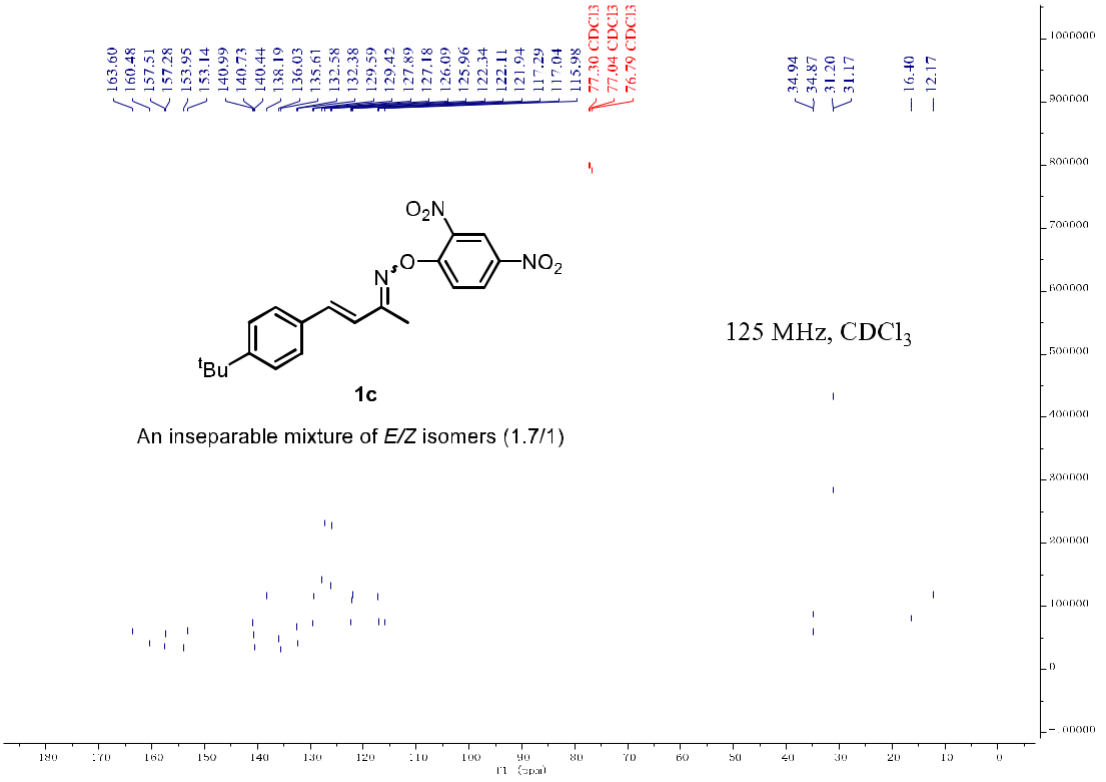
2E,3E)-4-(p-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1b)


8.95
8.94
8.89
8.89
8.45
8.44
8.44
8.43
8.42
8.42
8.05
8.04
8.03
8.02
7.73
7.70
7.51
7.50
7.43
7.42
7.26
7.24
7.22
7.20
7.16
7.15
7.13
7.11
6.90
6.87
2.41
2.39
2.38
2.32
O2N
NO2
N
O
Me
1b
500 MHz, CDCl3
An inseparable mixture of E/Z isomers (1.7/1)
0.52
0.92
1.60
1.57
0.60
1.20
2.05
3.25
1.60
1.00
3.01
4.80
1.73
f1 (ppm)

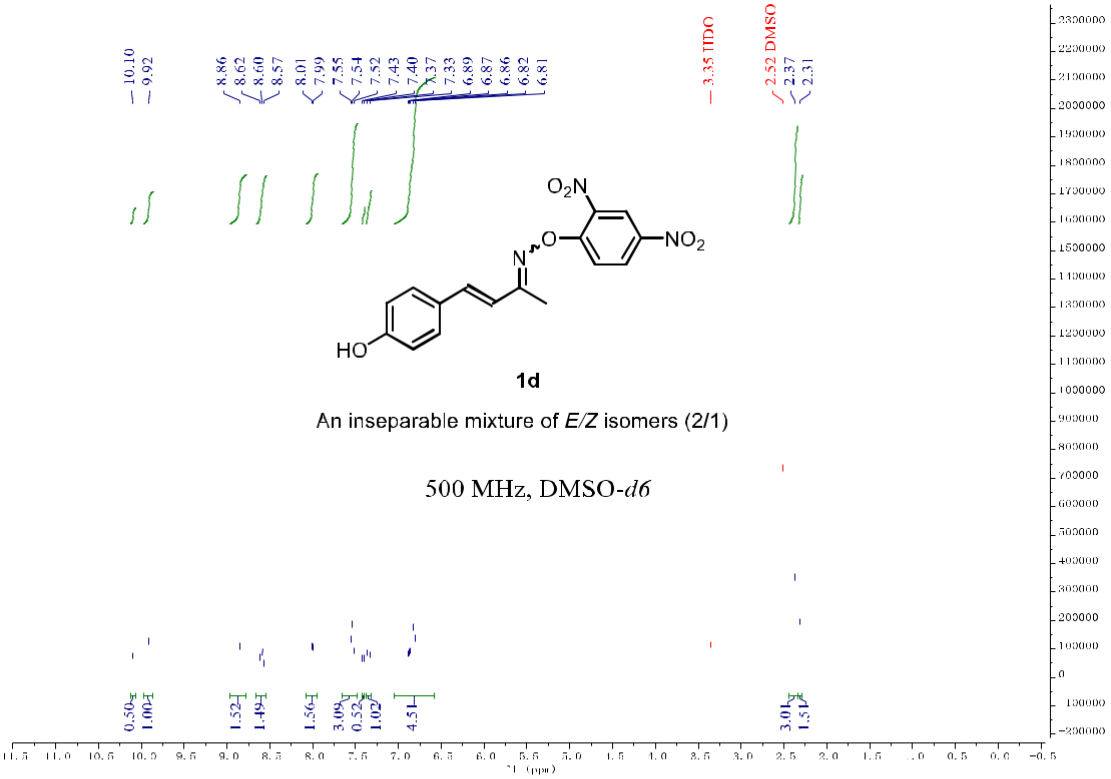

163.56
160.45
157.52
157.29
141.15
140.83
140.72
140.44
139.95
138.28
136.02
135.59
132.60
132.37
129.84
129.71
129.60
129.43
128.04
127.32
122.34
122.11
121.77
117.26
117.07
115.71
77.30 CDCl3
77.04 CDCl3
76.79 CDCl3
21.51
21.42
16.41
12.16
O2N
NO2
N
O
Me
1b
125 MHz, CDCl3
An inseparable mixture of E/Z isomers (1.7/1)
f1 (ppm)

(2E,3E)-4-(4-(tert-butyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1c)





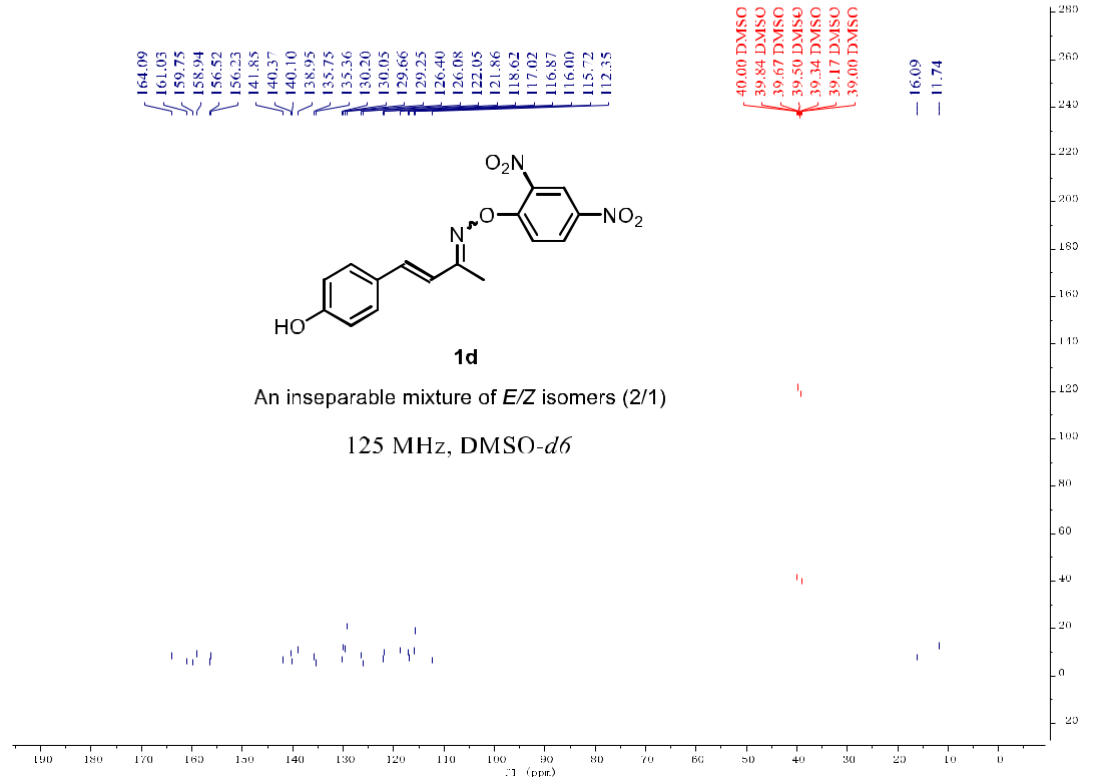
(2E,3E)-4-(4-hydroxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1d)



1d

An inseparable mixture of *E*/*Z* isomers (2/1)

500 MHz, DMSO-*d6*

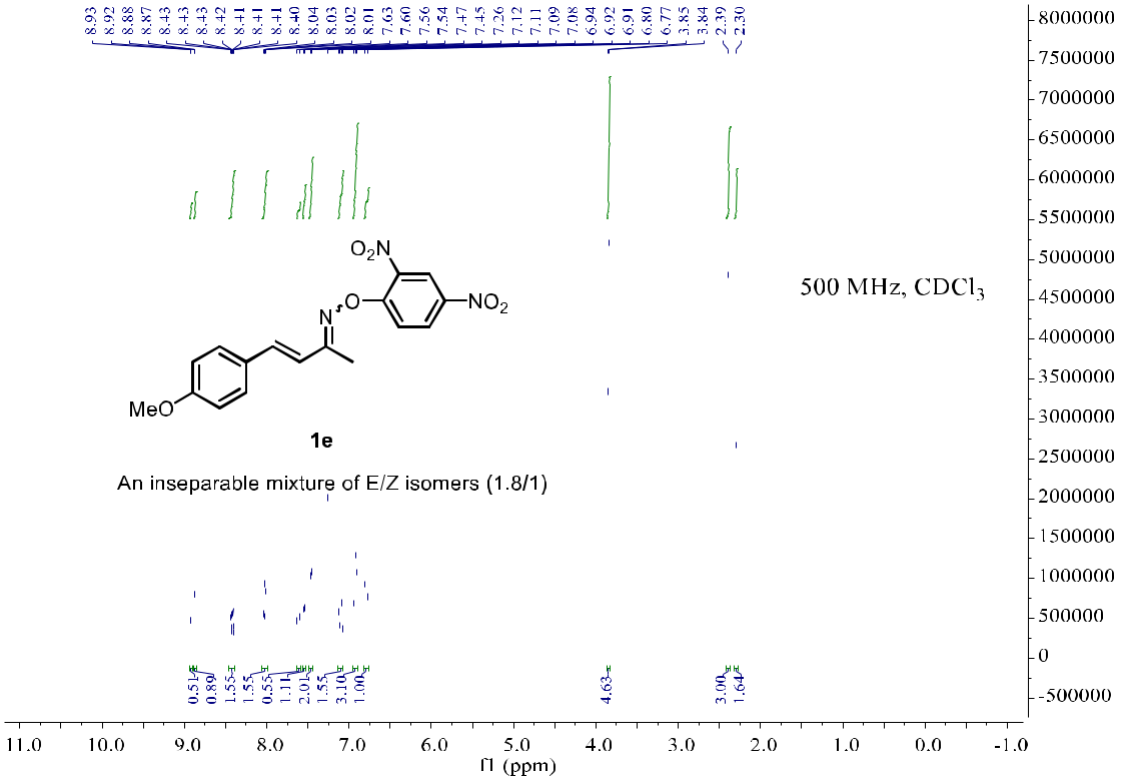


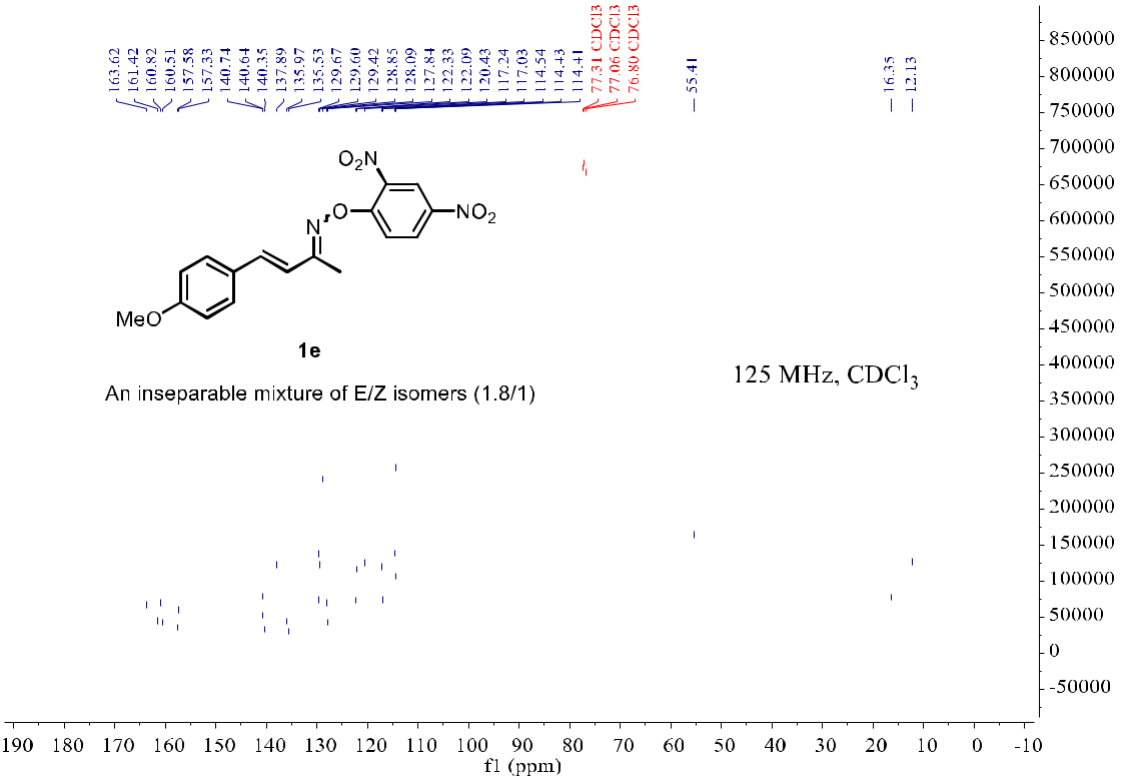
1d

An inseparable mixture of *E*/*Z* isomers (2/1)

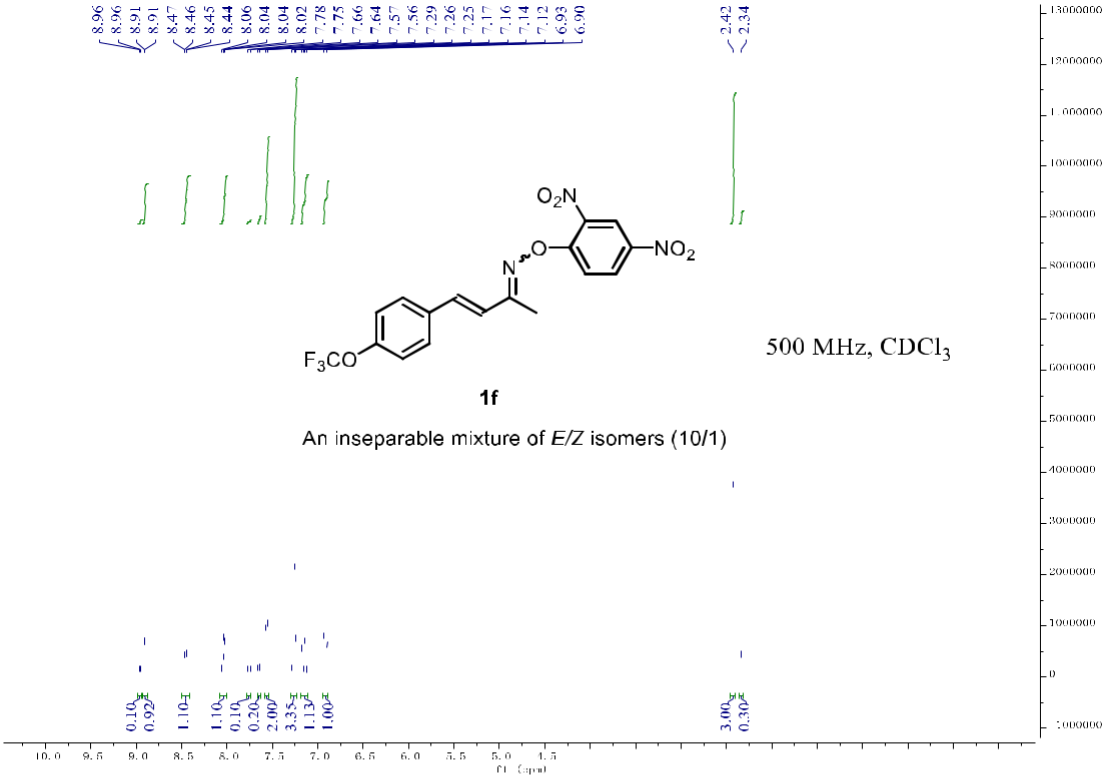
125 MHz, DMSO-*d6*

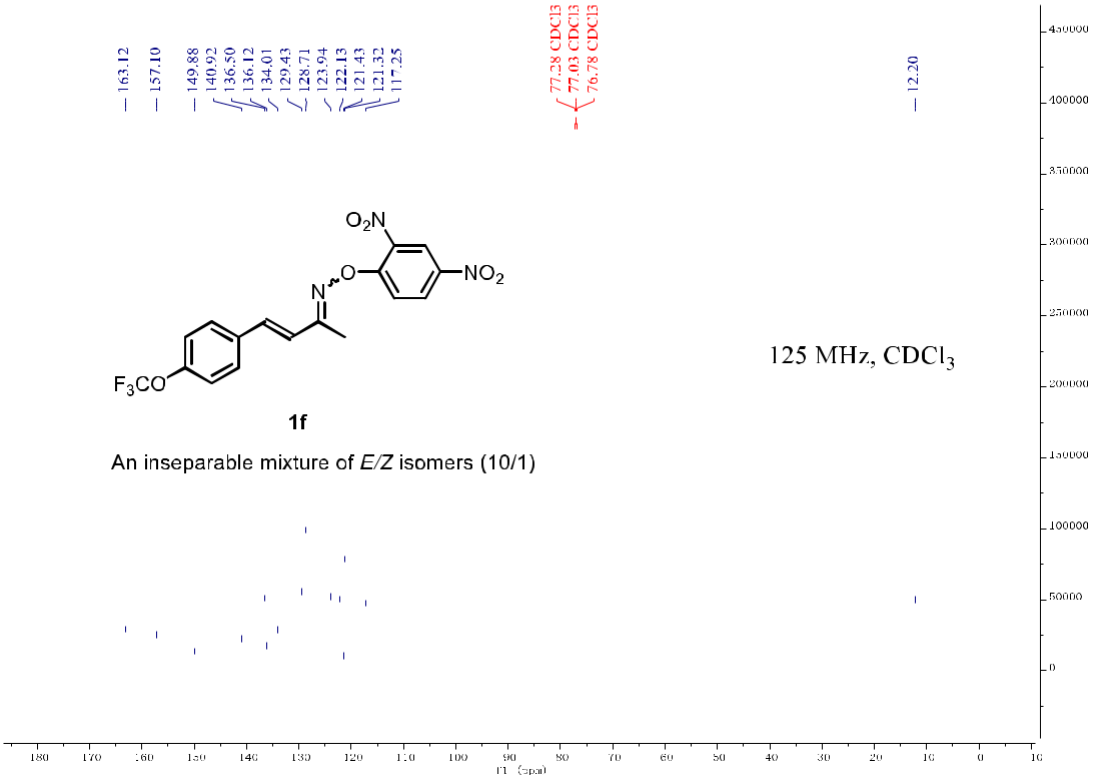
(2E,3E)-4-(4-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1e)



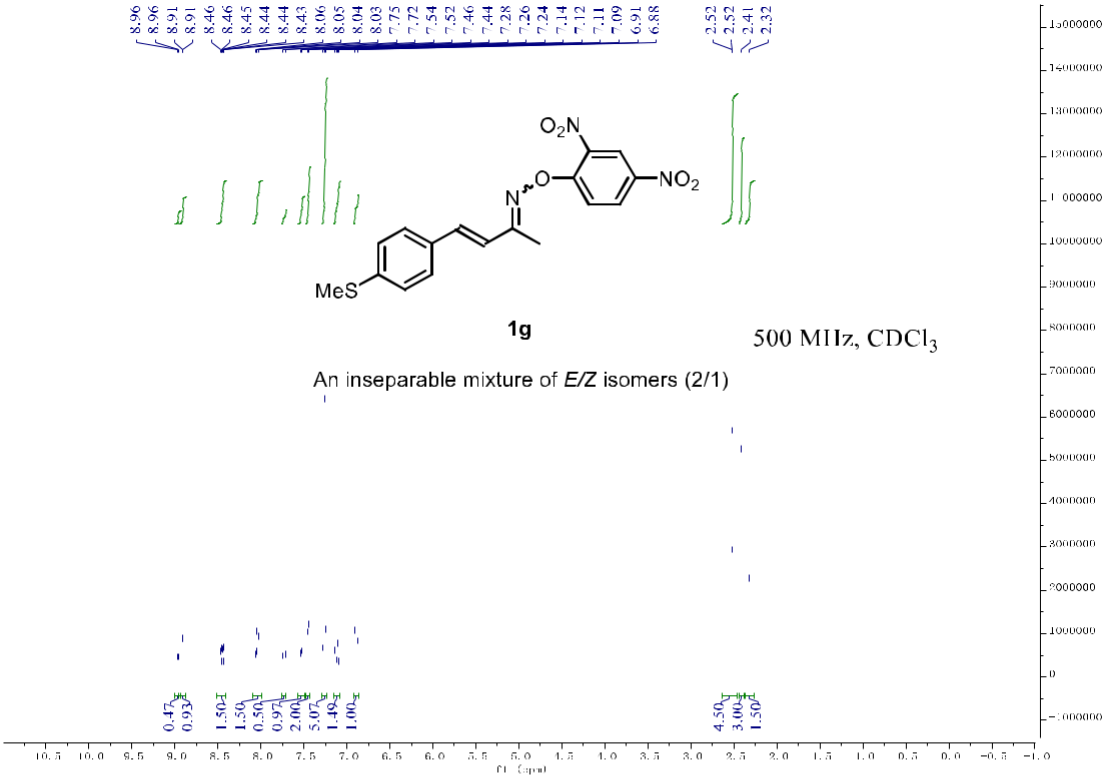


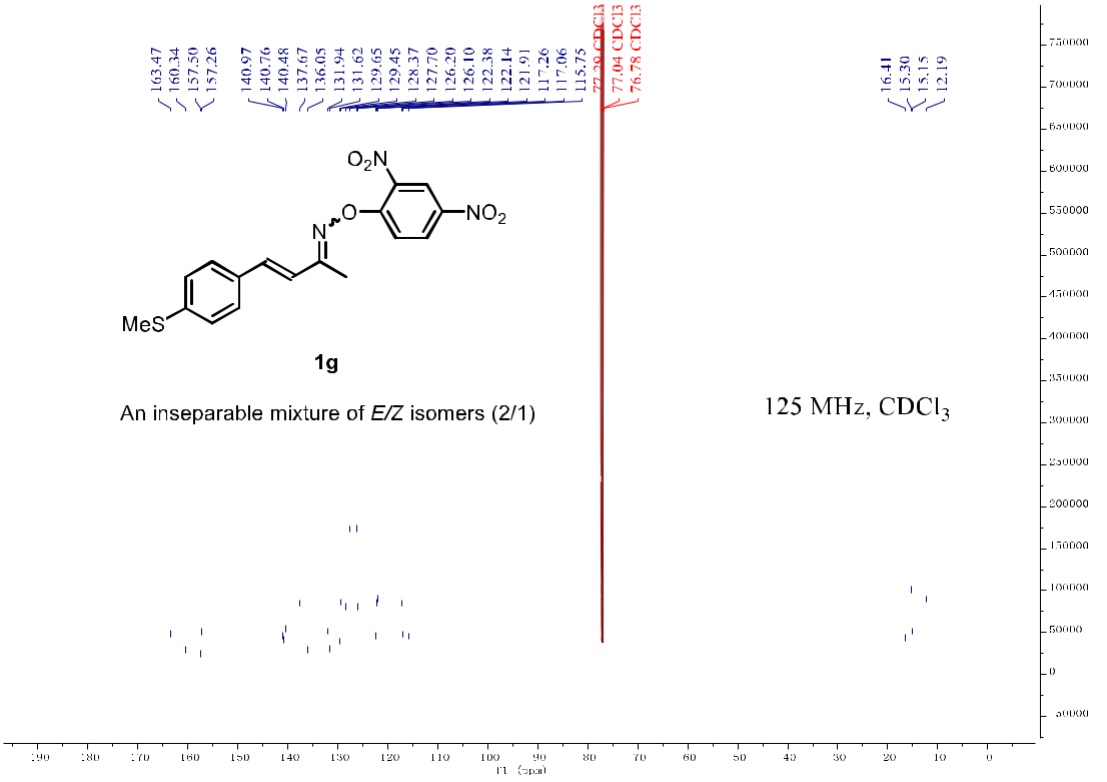
(2E,3E)-4-(4-(trifluoromethoxy)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1f)



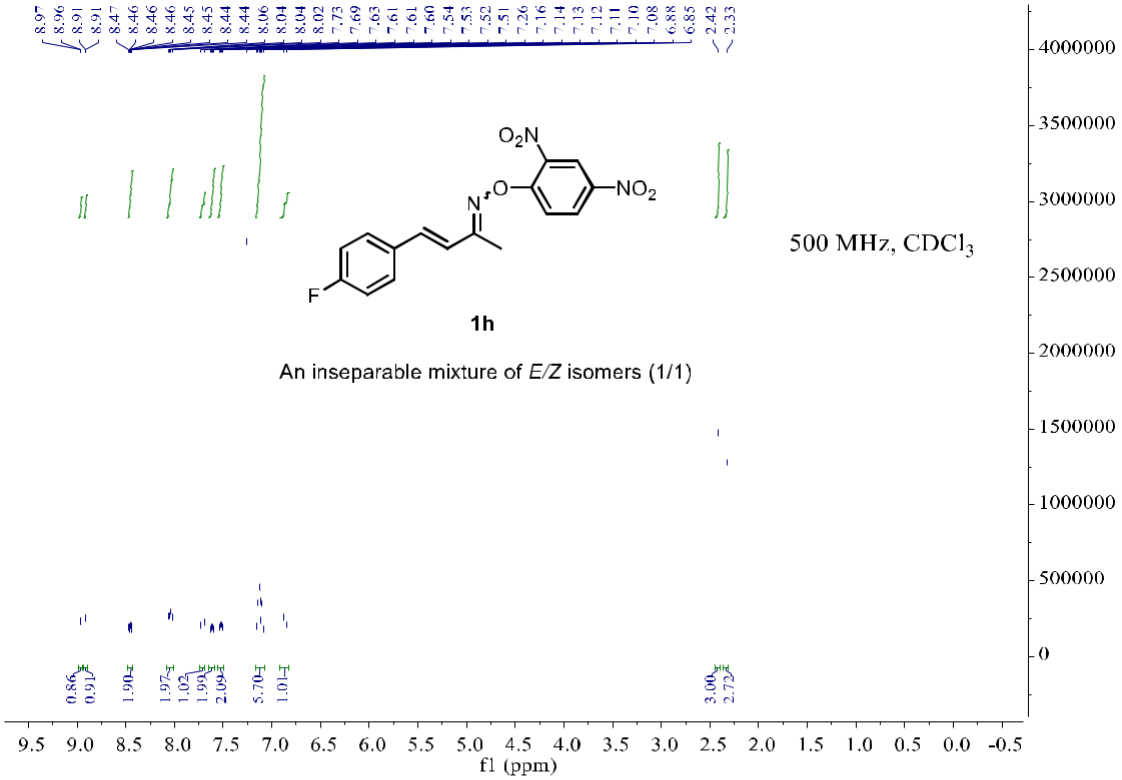


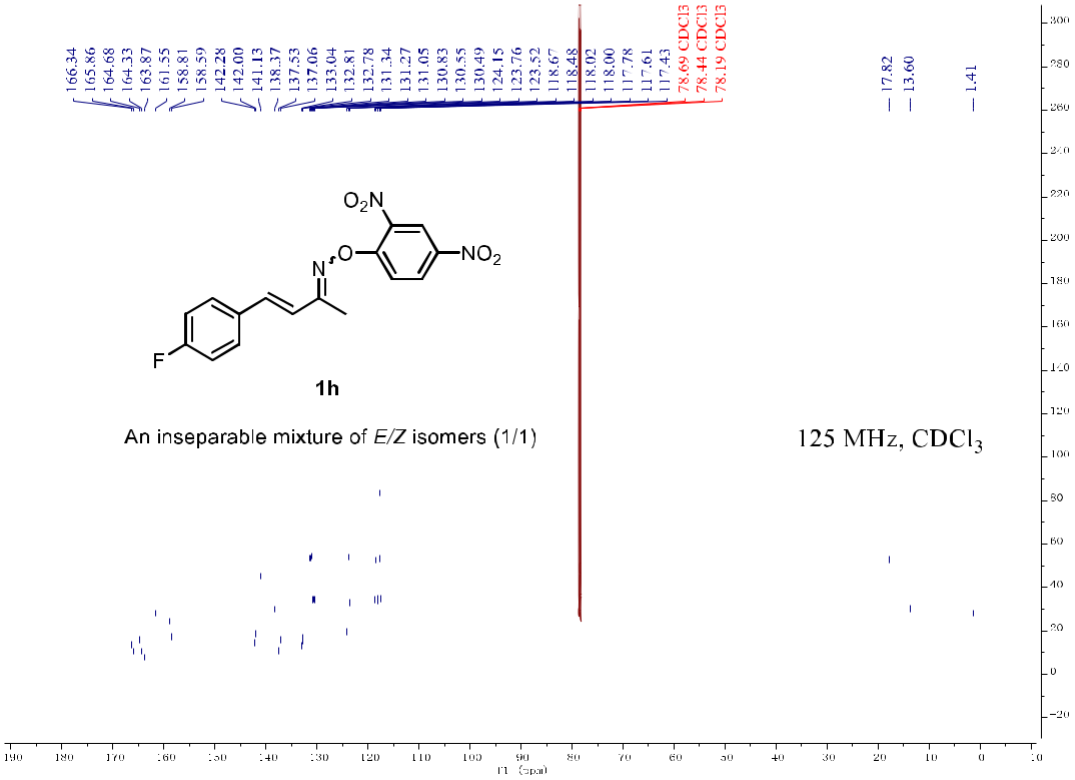
(2E,3E)-4-(4-(methylthio)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1g)



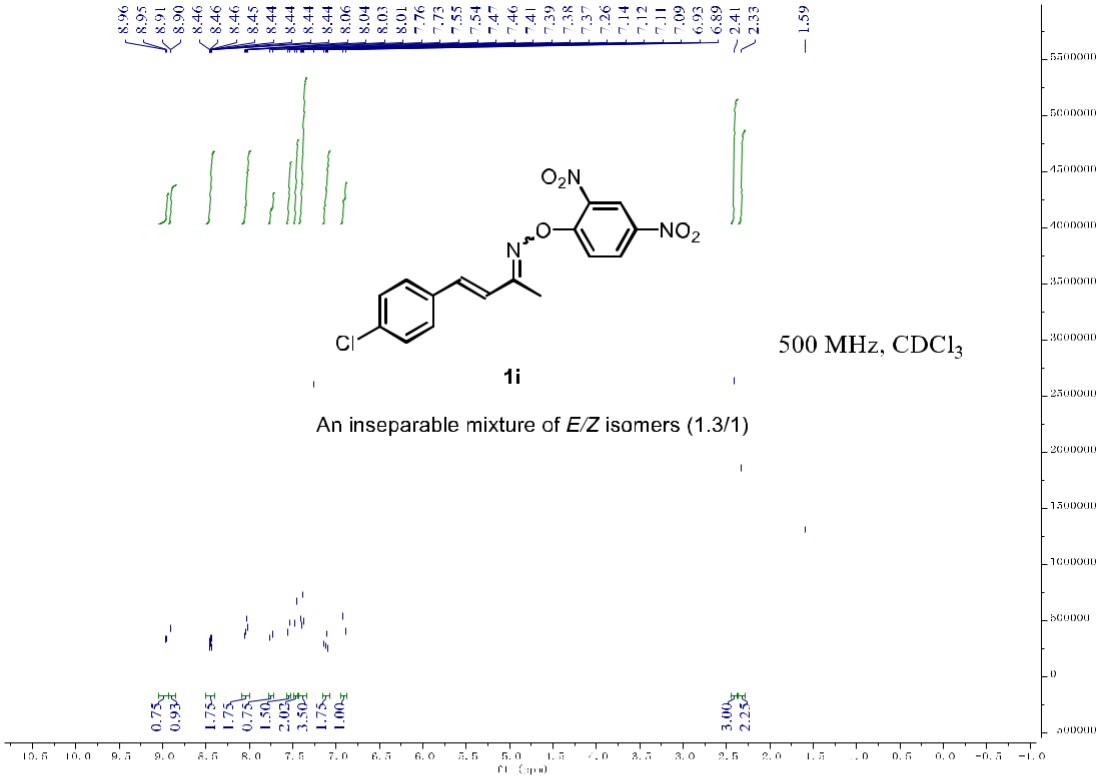


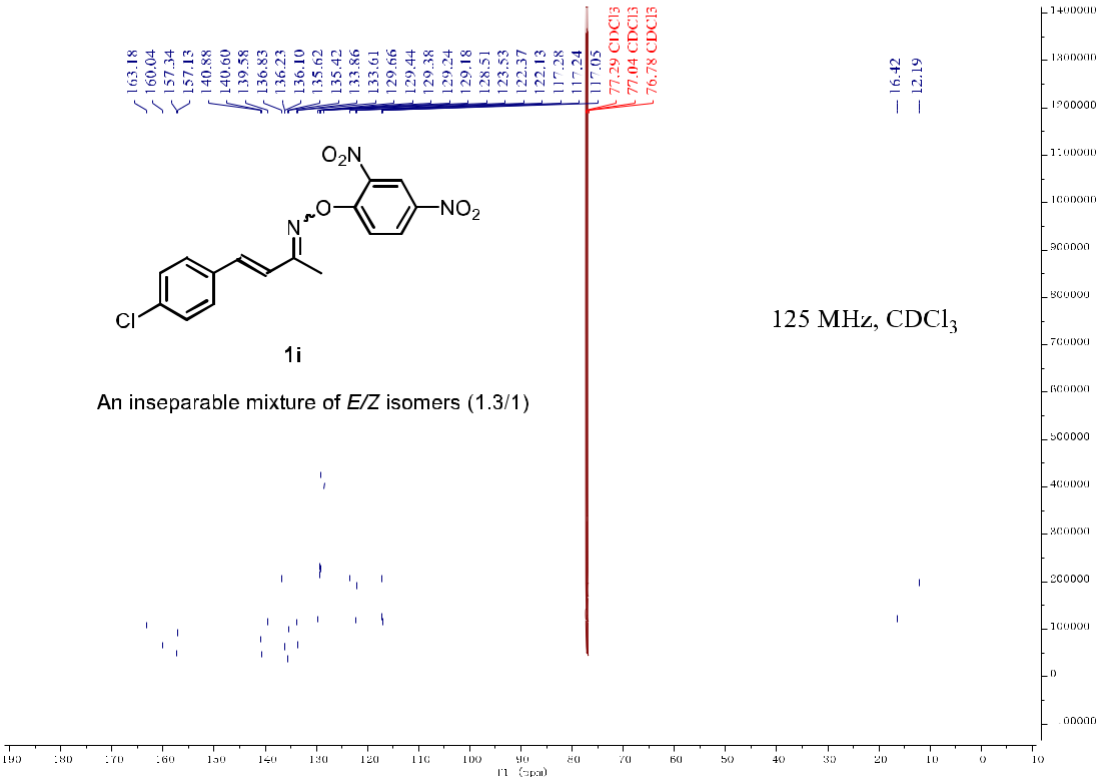
(2E,3E)-4-(4-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1h)



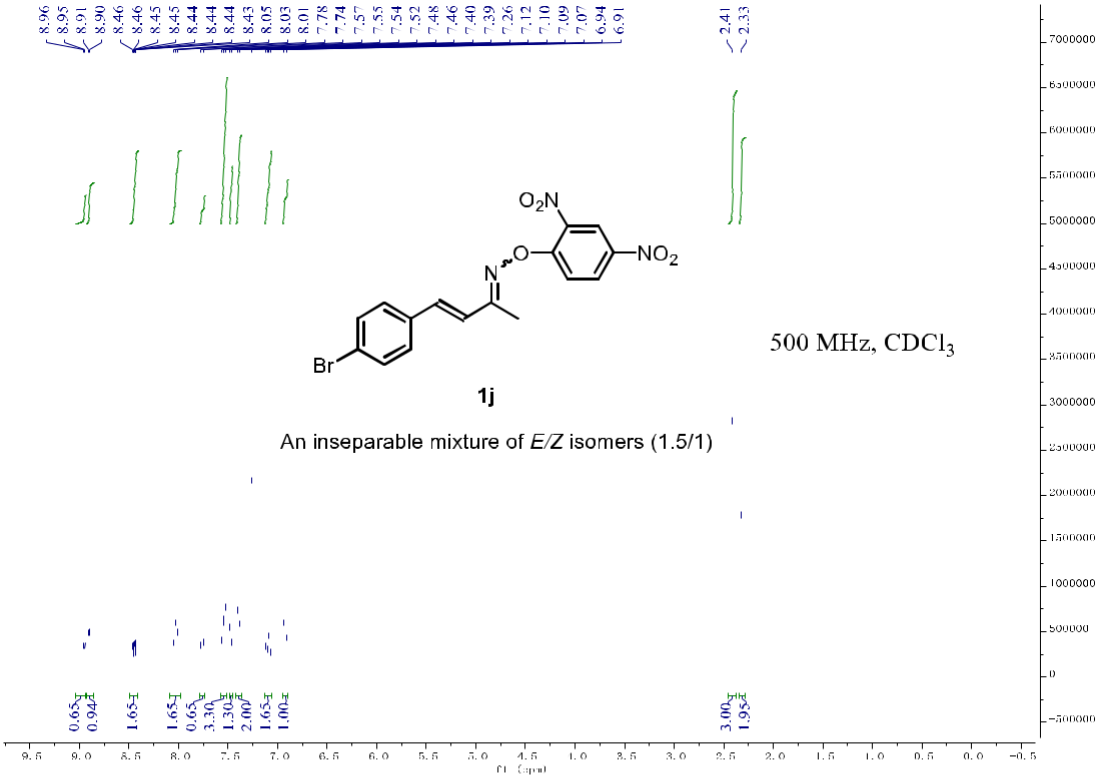


(2E,3E)-4-(4-chlorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1i)





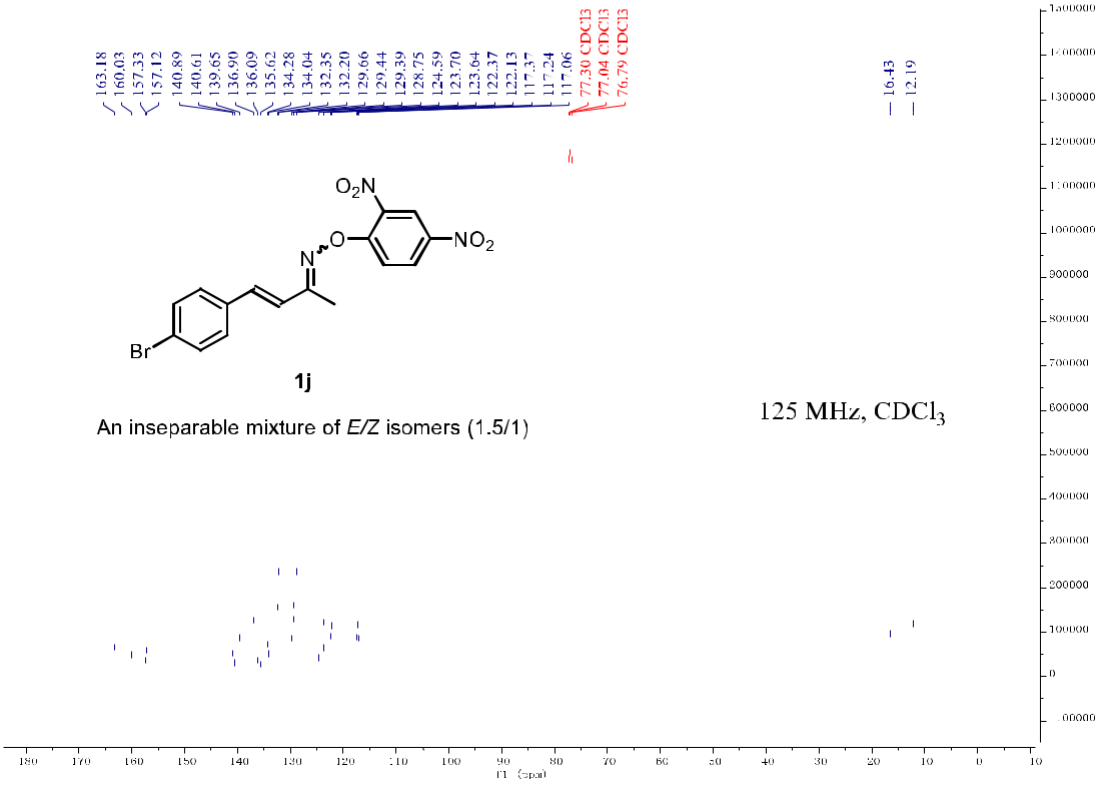
(2E,3E)-4-(4-bromophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1j)



1j

An inseparable mixture of *E*/*Z* isomers (1.5/1)

500 MHz, $CDCl_3$

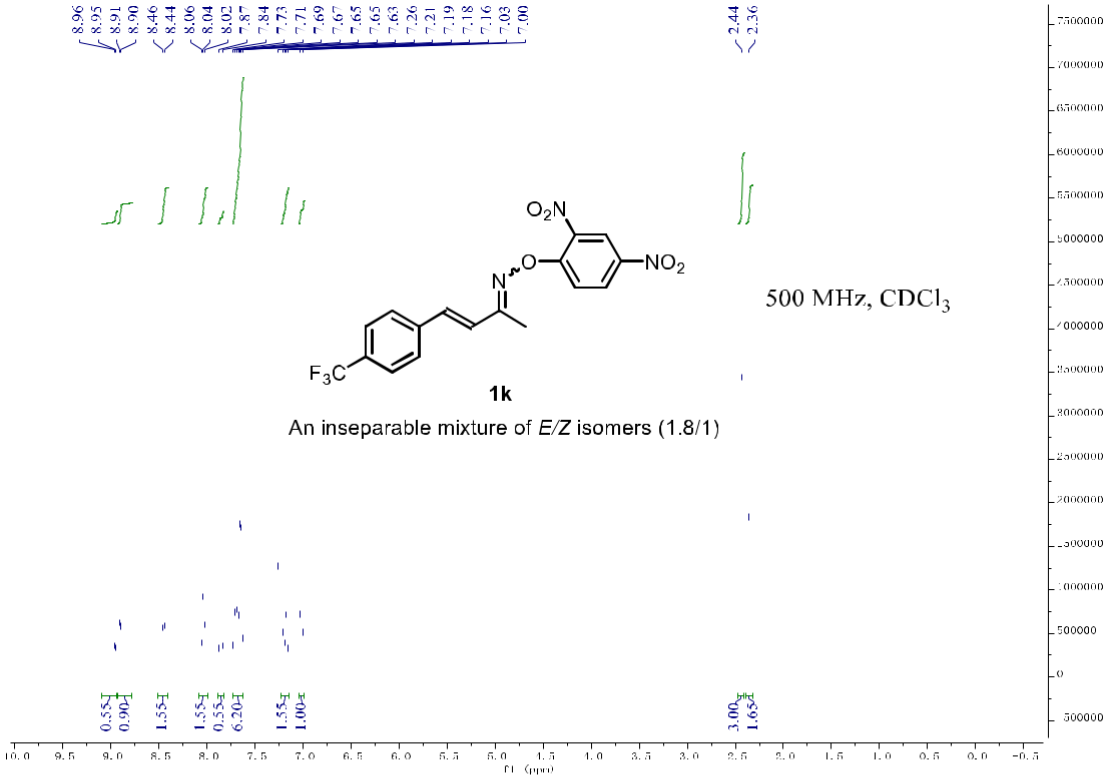


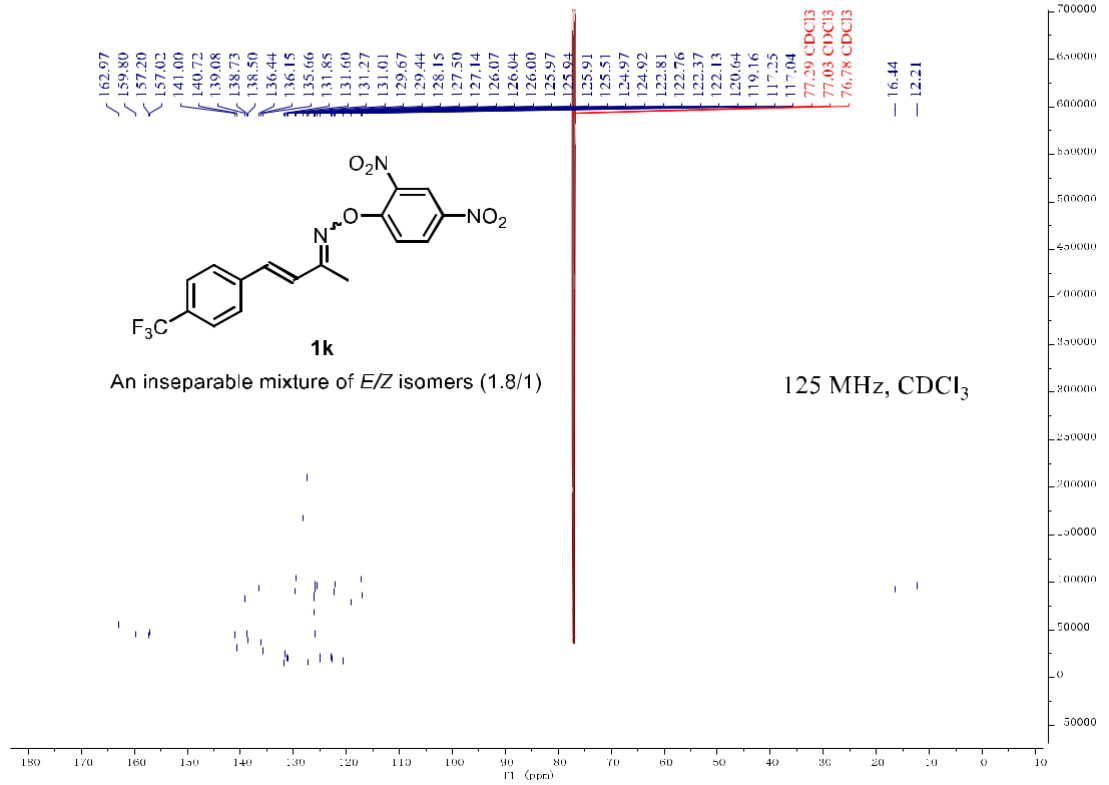
1j

An inseparable mixture of *E*/*Z* isomers (1.5/1)

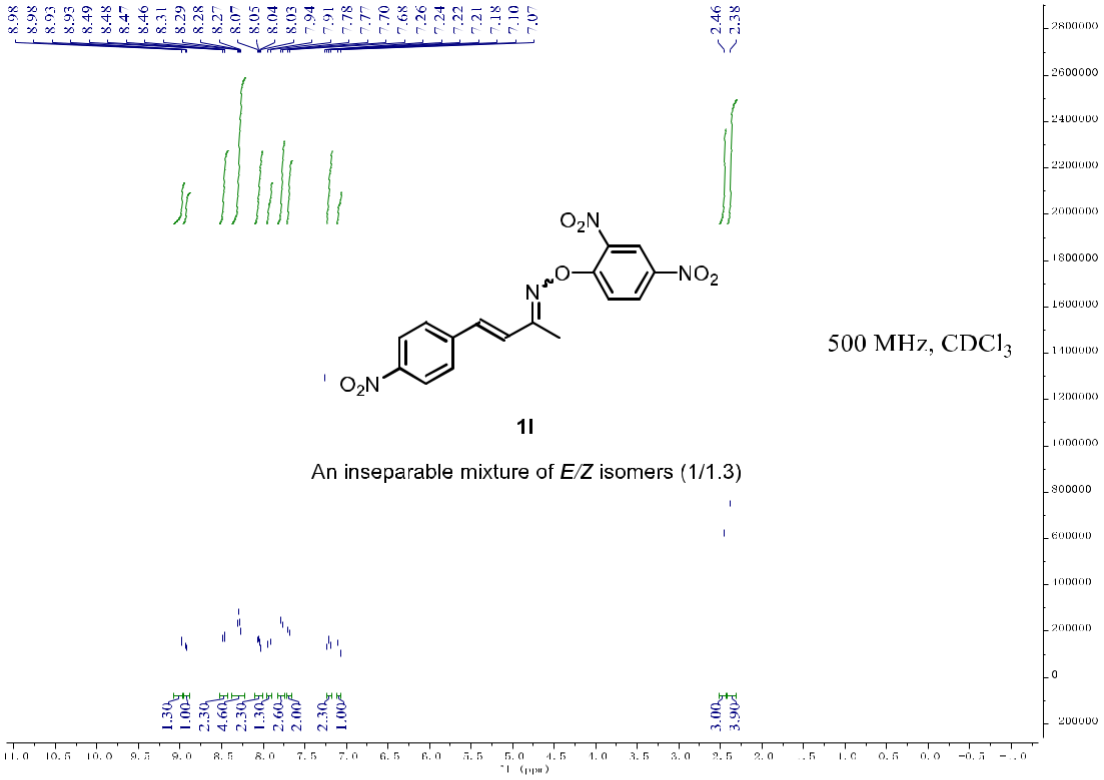
125 MHz, $CDCl_3$

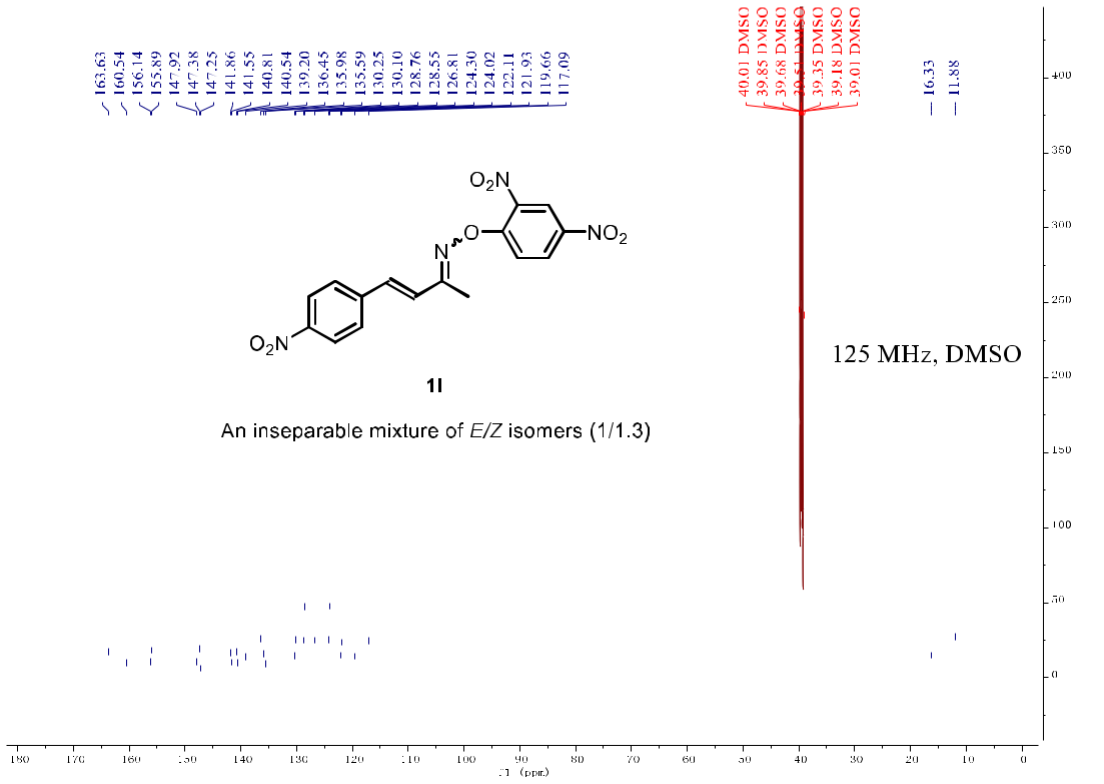
(2E,3E)-4-(4-(trifluoromethyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1k)



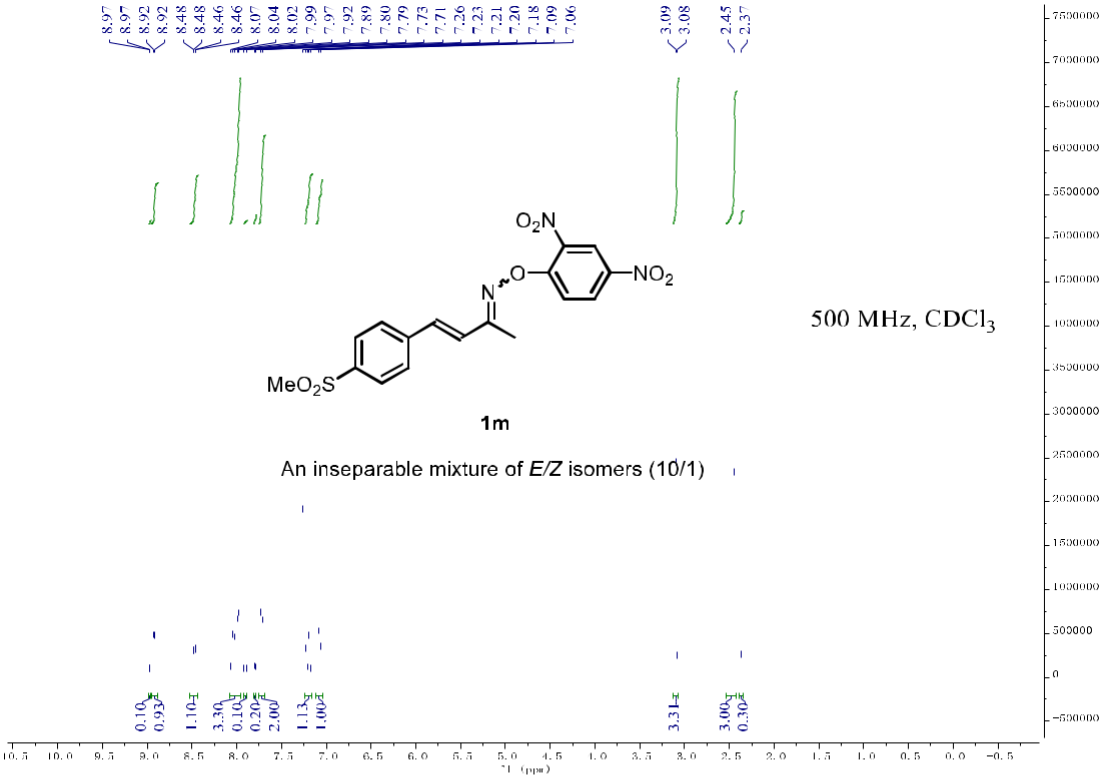


(2E,3E)-4-(4-nitrophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1l)



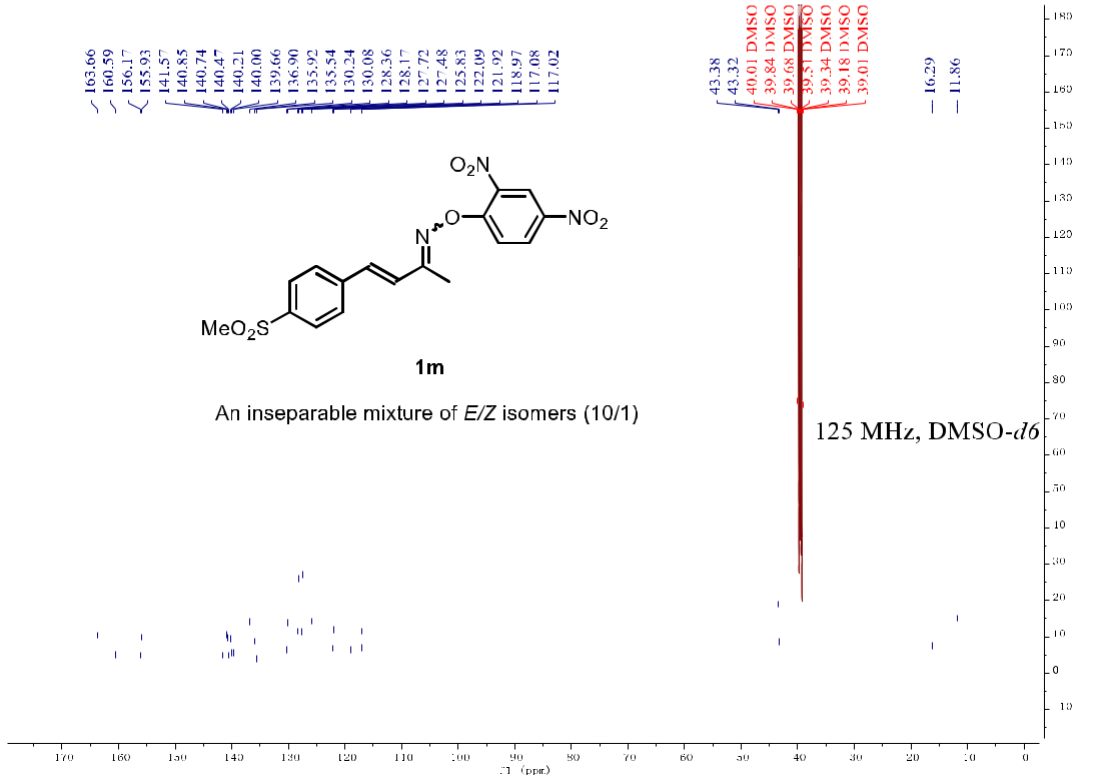


(2E,3E)-4-(4-(methylsulfonyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1m)



1m

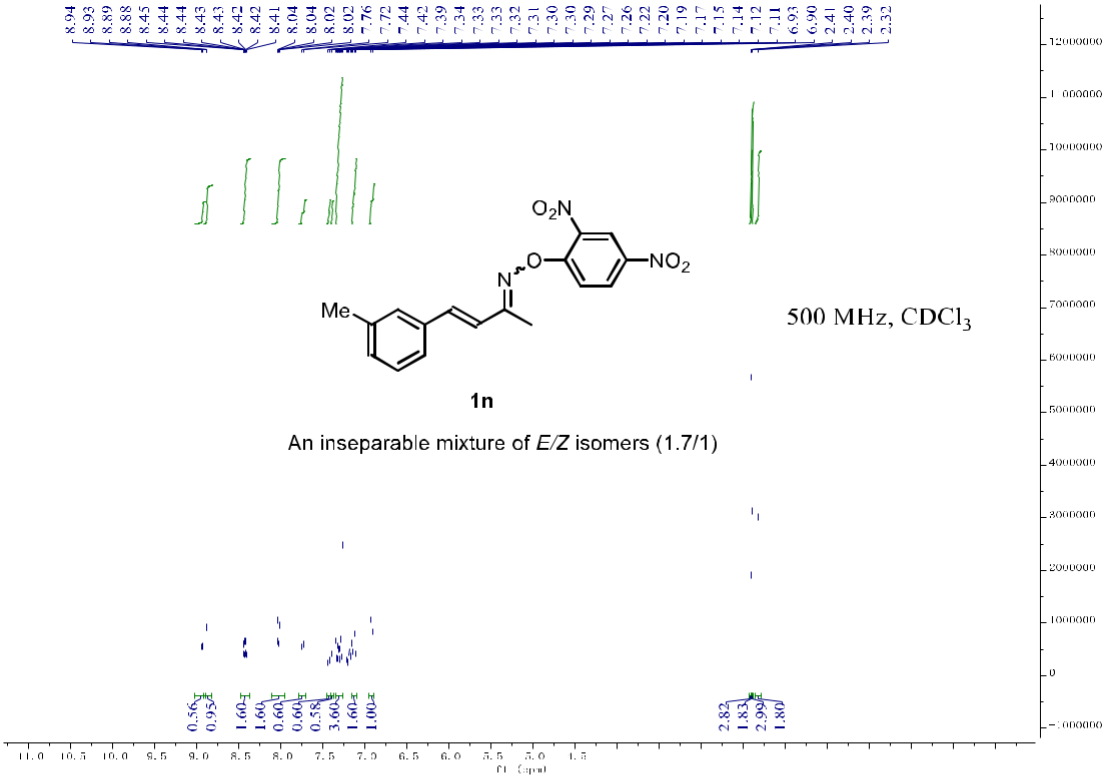
An inseparable mixture of *E*/*Z* isomers (10/1)

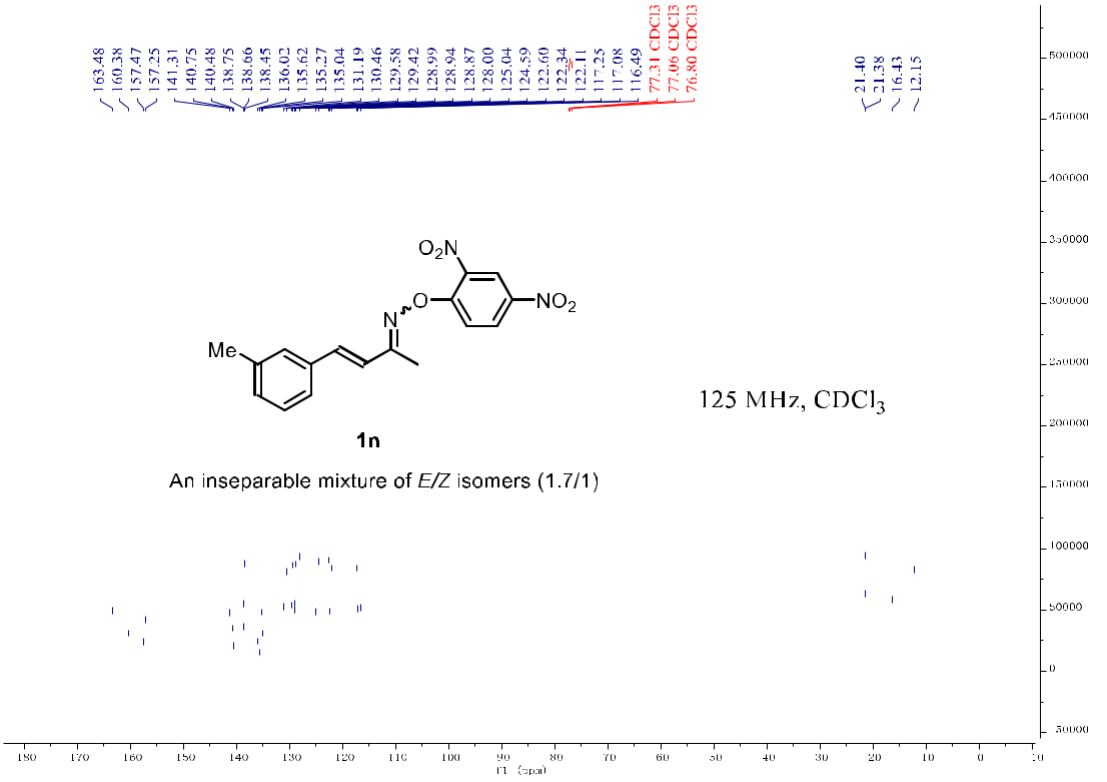


1m

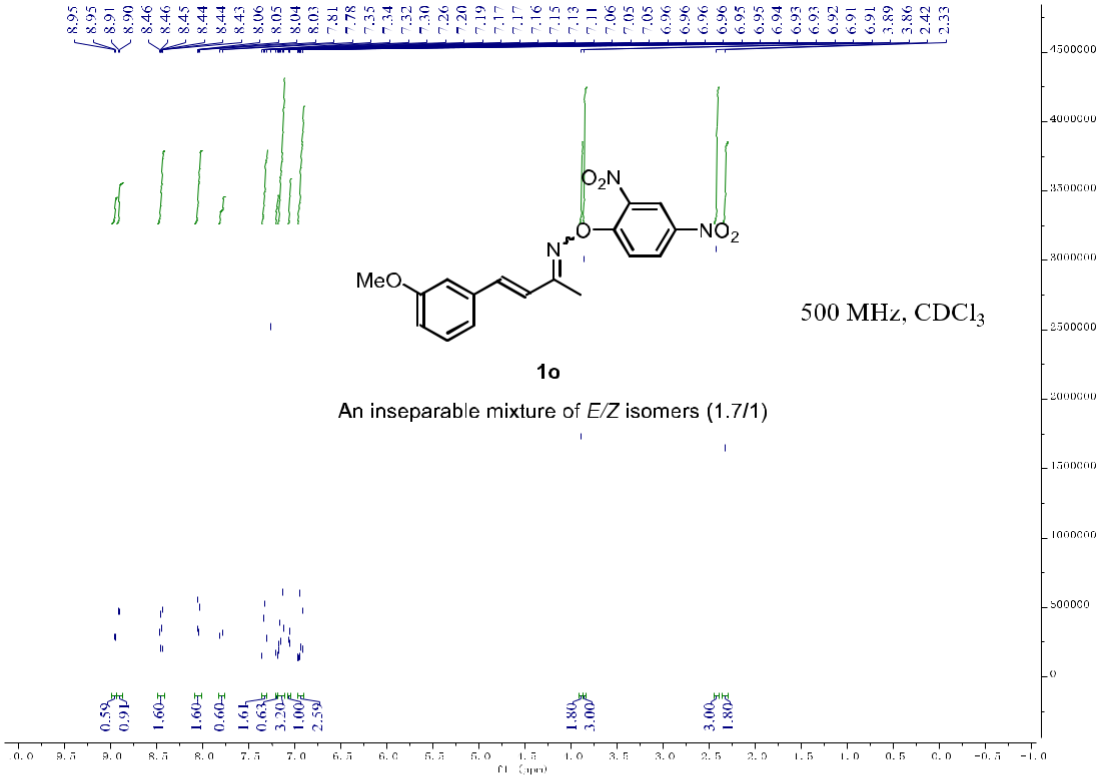
An inseparable mixture of *E*/*Z* isomers (10/1)

(2E,3E)-4-(m-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1n)



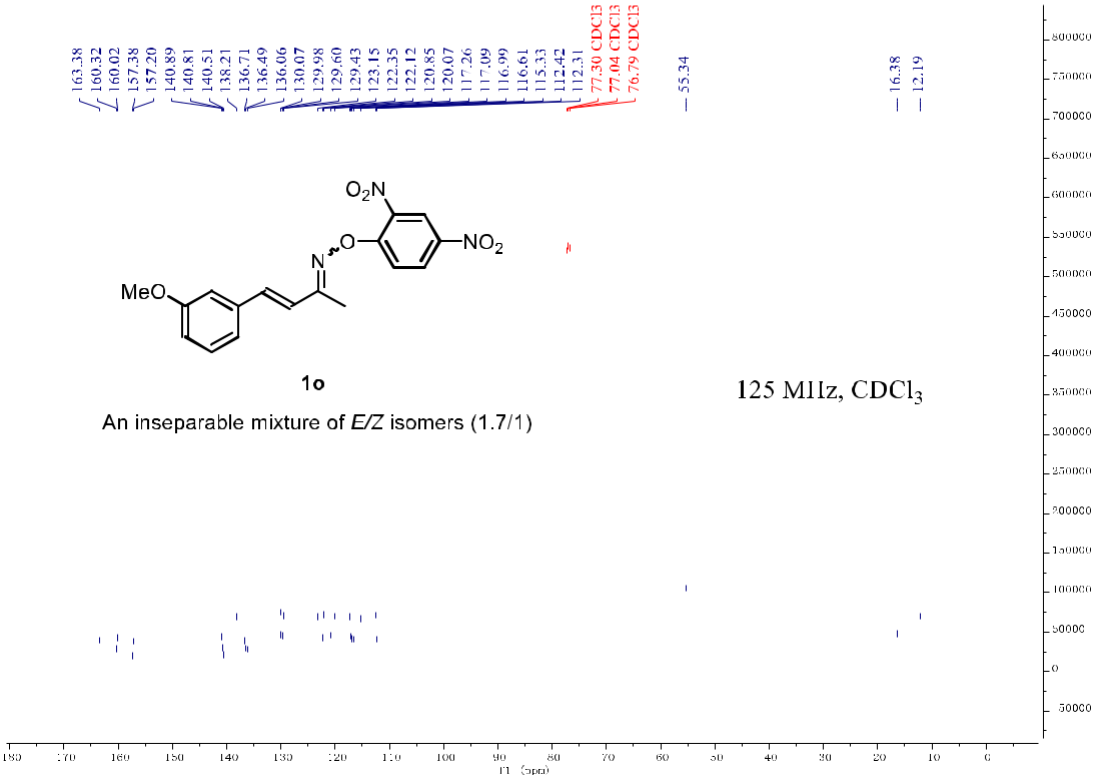


(2E,3E)-4-(3-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1o)



1o

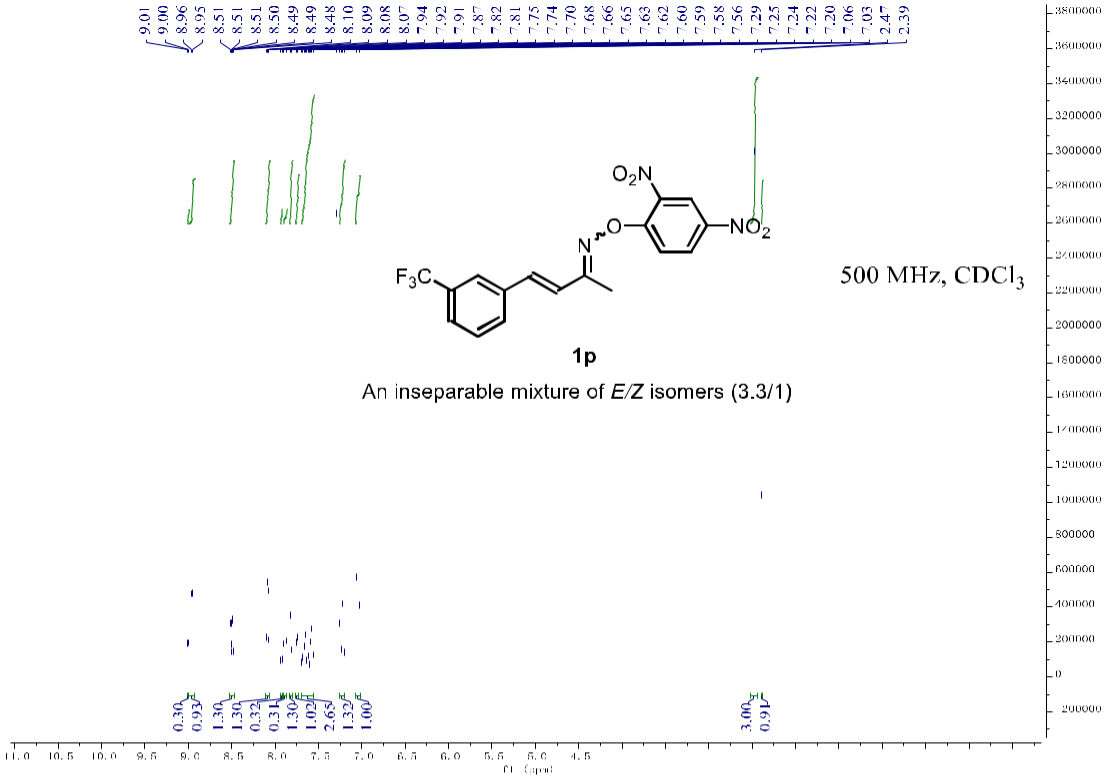
An inseparable mixture of *E/Z* isomers (1.7/1)

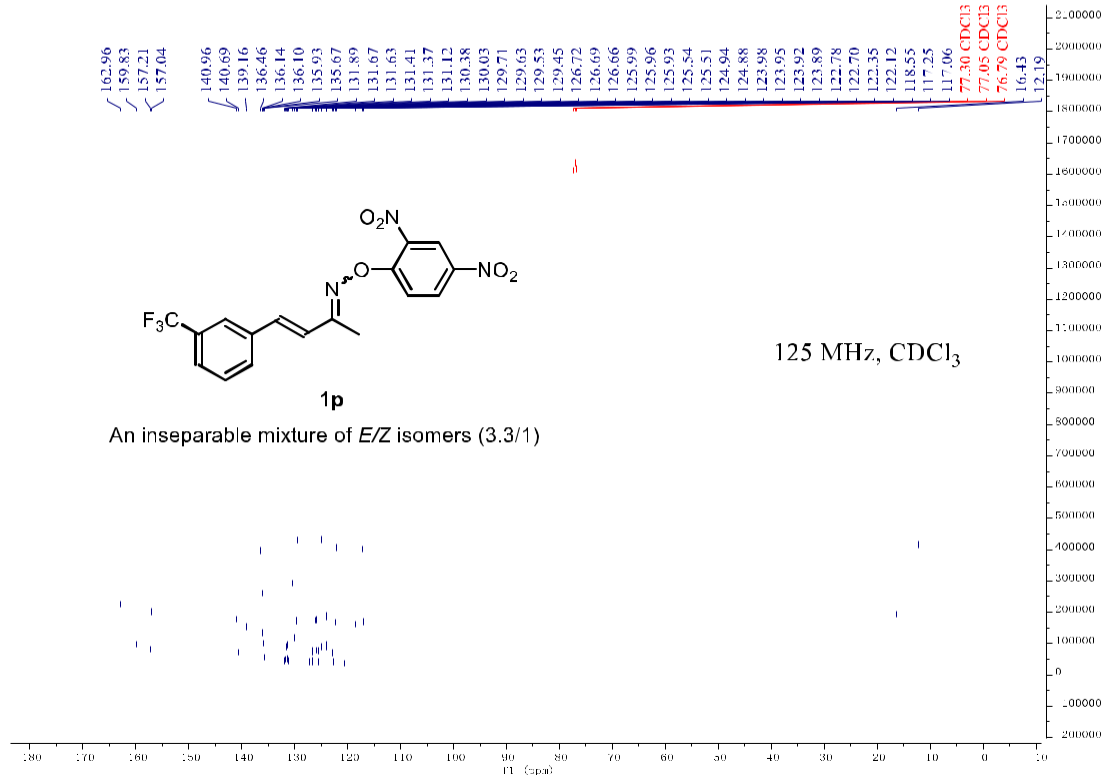


1o

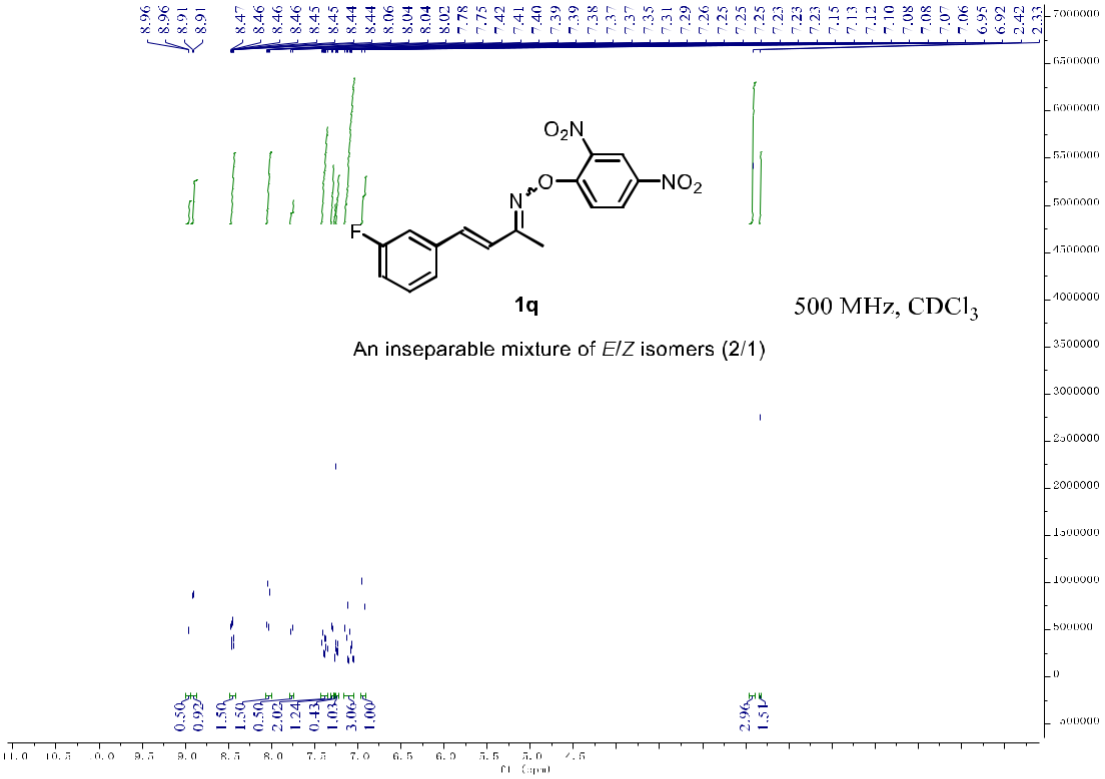
An inseparable mixture of *E/Z* isomers (1.7/1)

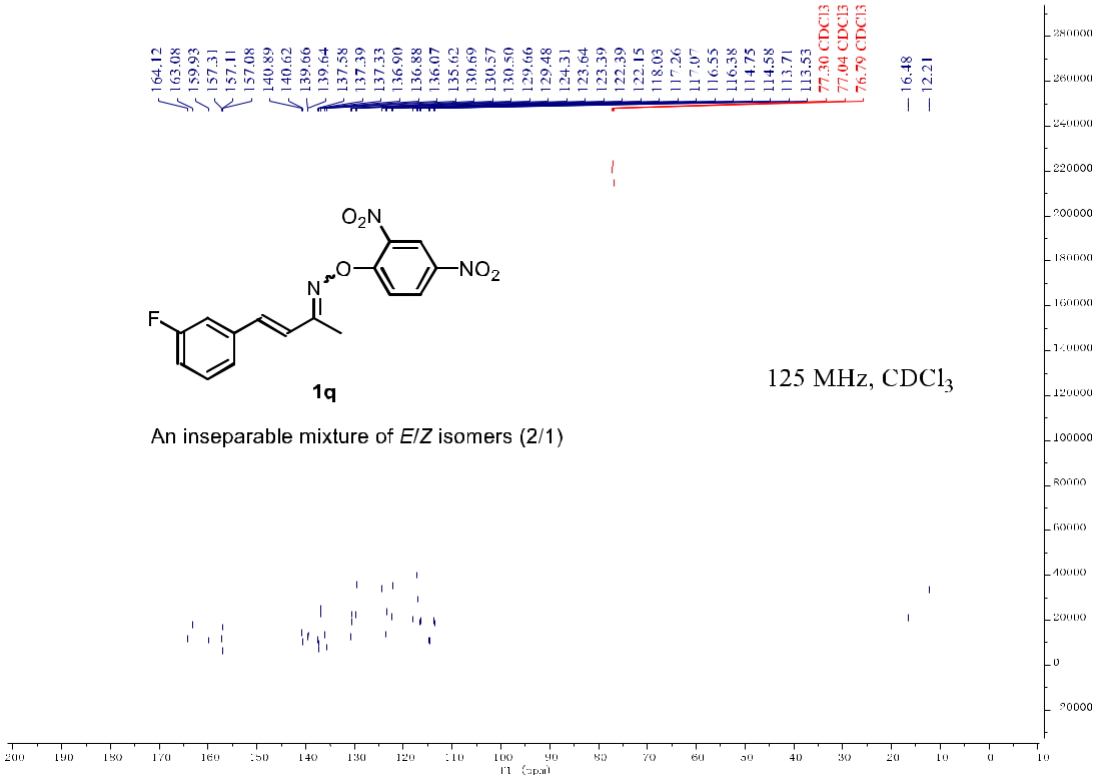
(2E,3E)-4-(3-(trifluoromethyl)phenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1p)



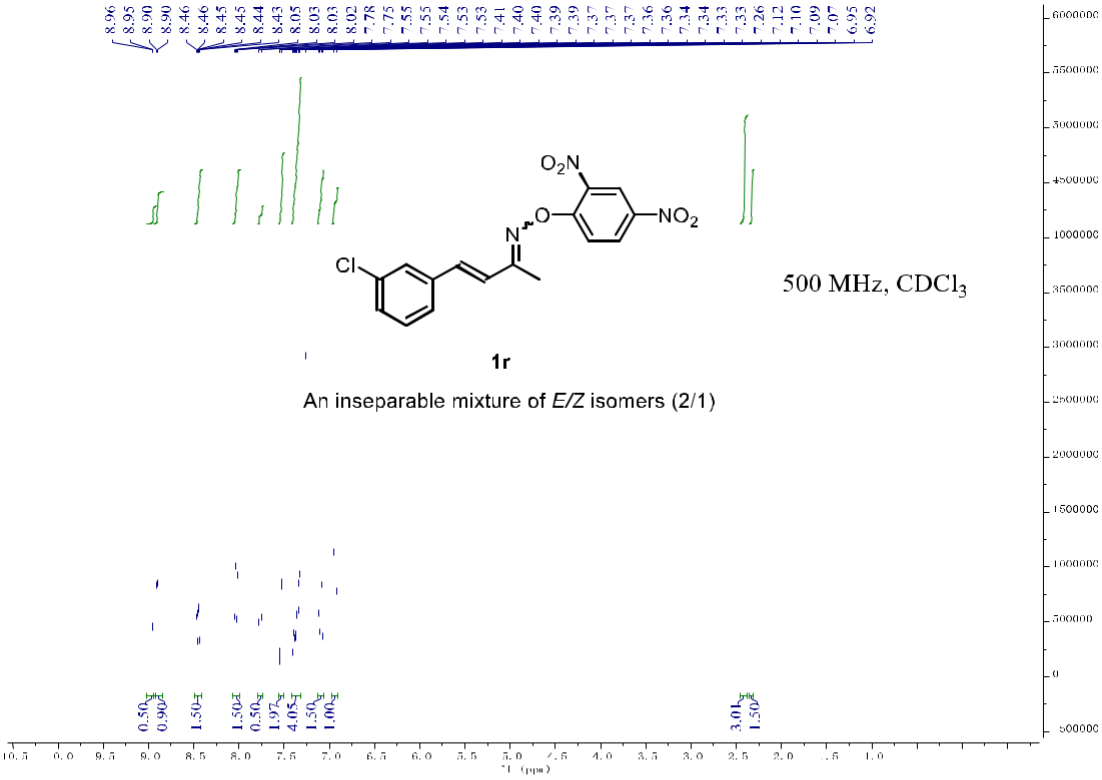


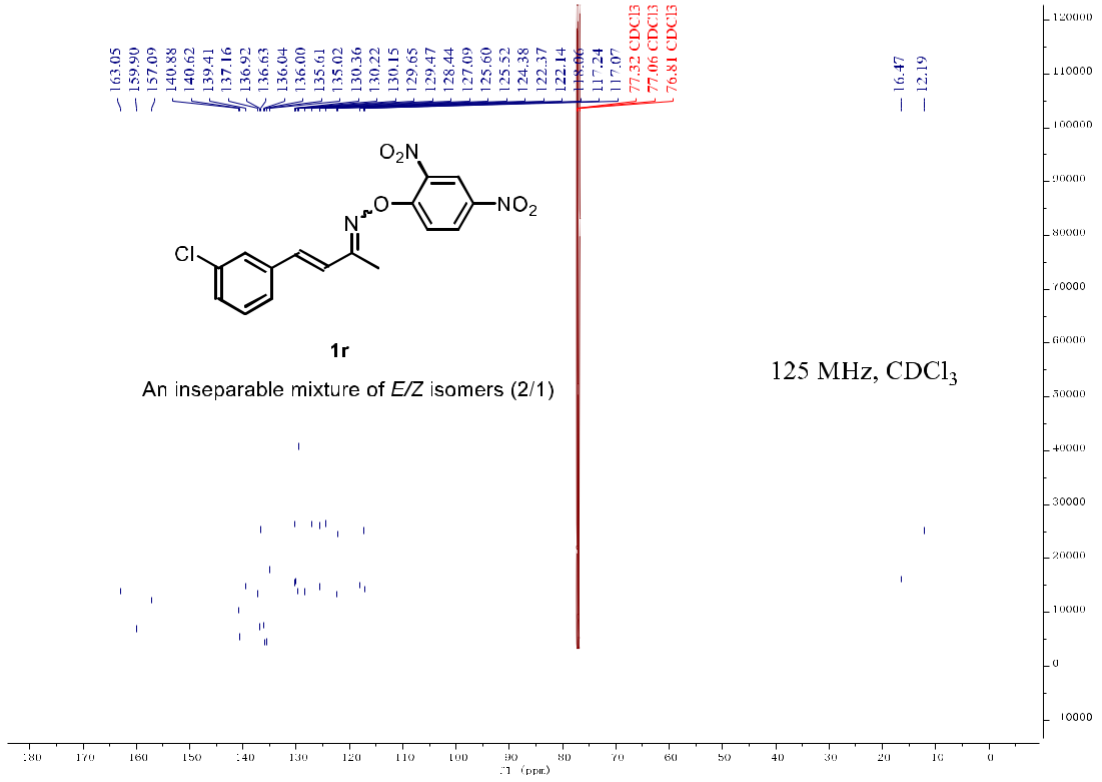
(2E,3E)-4-(3-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1q)



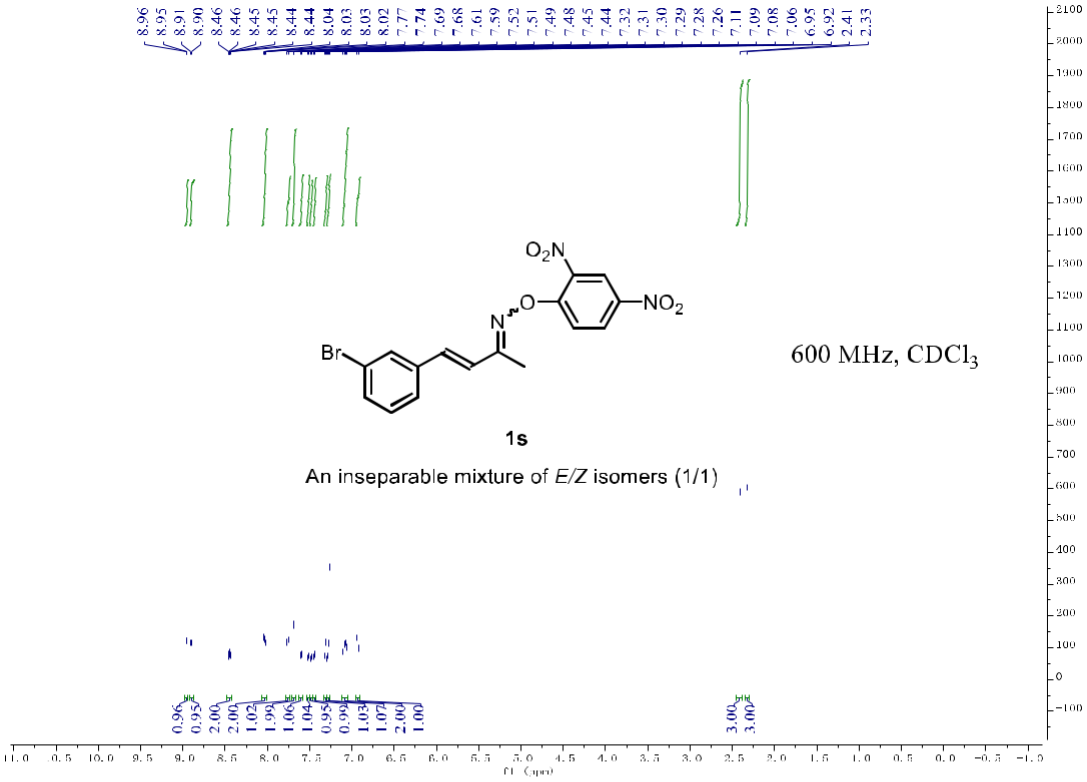


(2E,3E)-4-(3-chlorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1r)





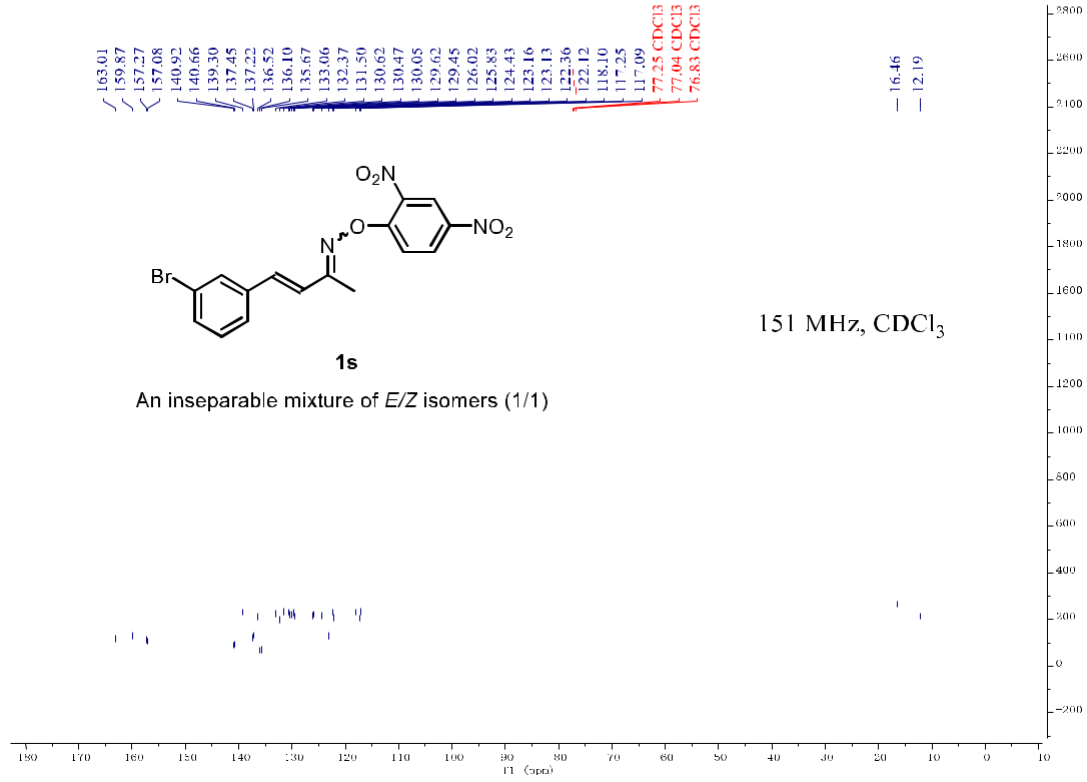
(2E,3E)-4-(3-bromophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1s)



600 MHz, $CDCl_3$

1s

An inseparable mixture of *E/Z* isomers (1/1)

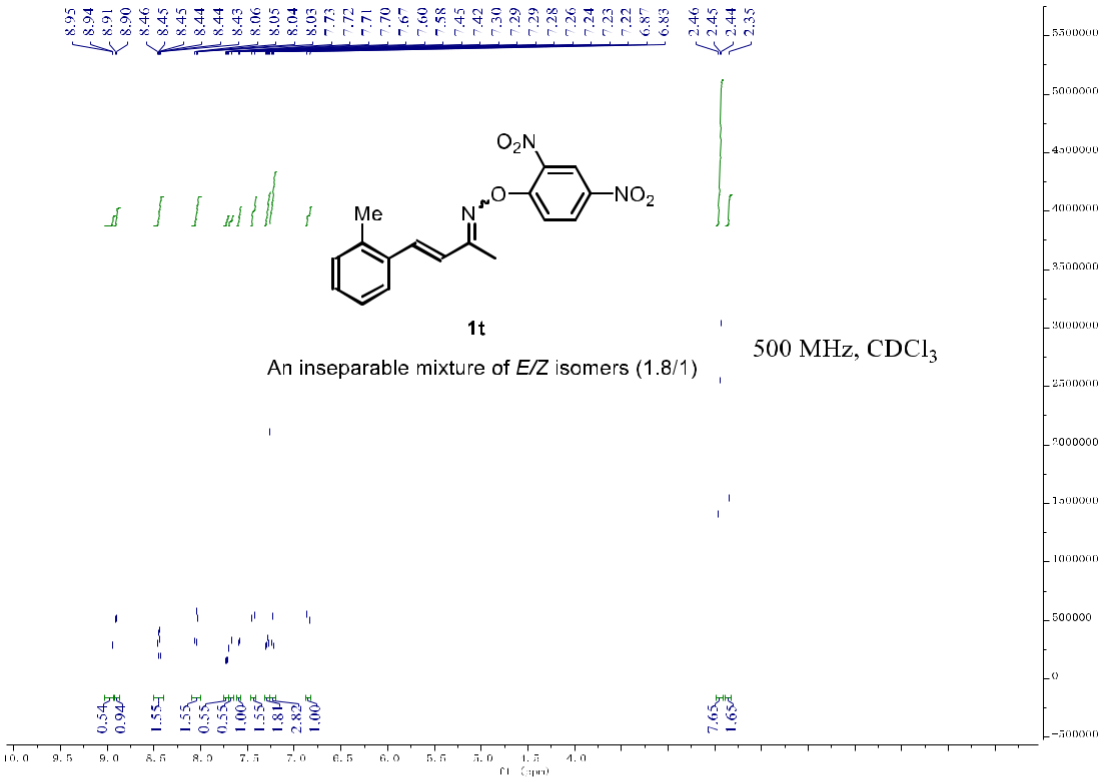


151 MHz, $CDCl_3$

1s

An inseparable mixture of *E/Z* isomers (1/1)

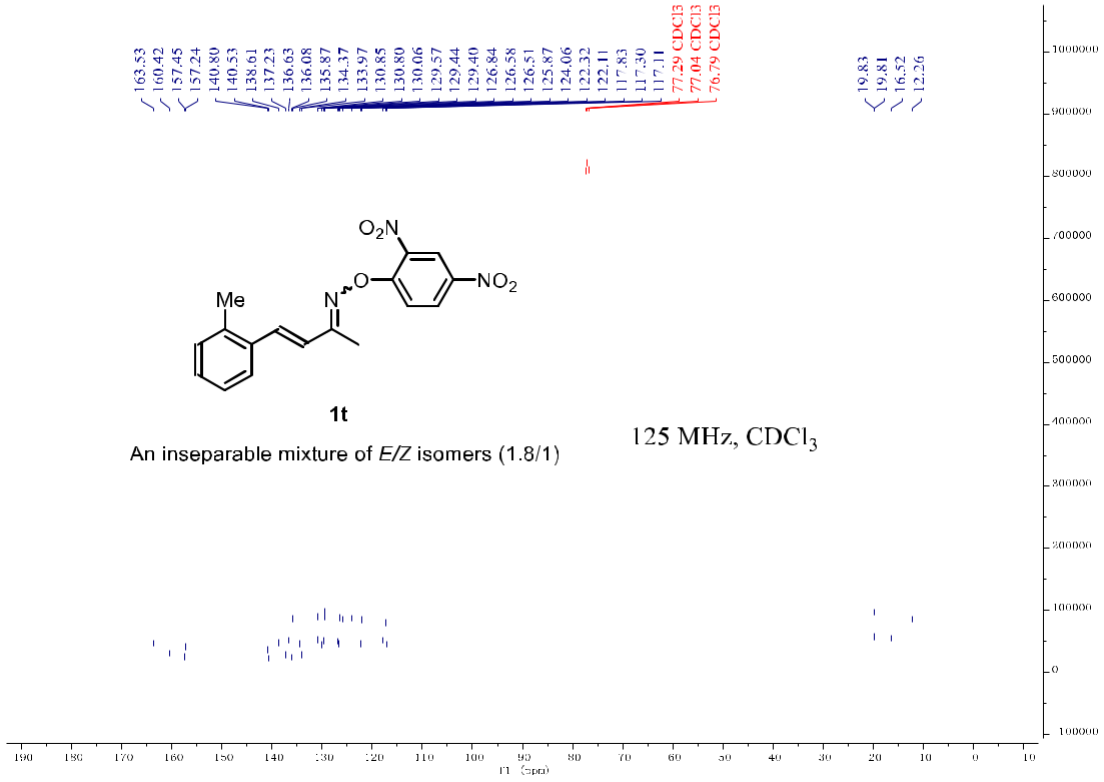
(2E,3E)-4-(o-tolyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1t)



1t

An inseparable mixture of *E/Z* isomers (1.8/1)

500 MHz, $CDCl_3$

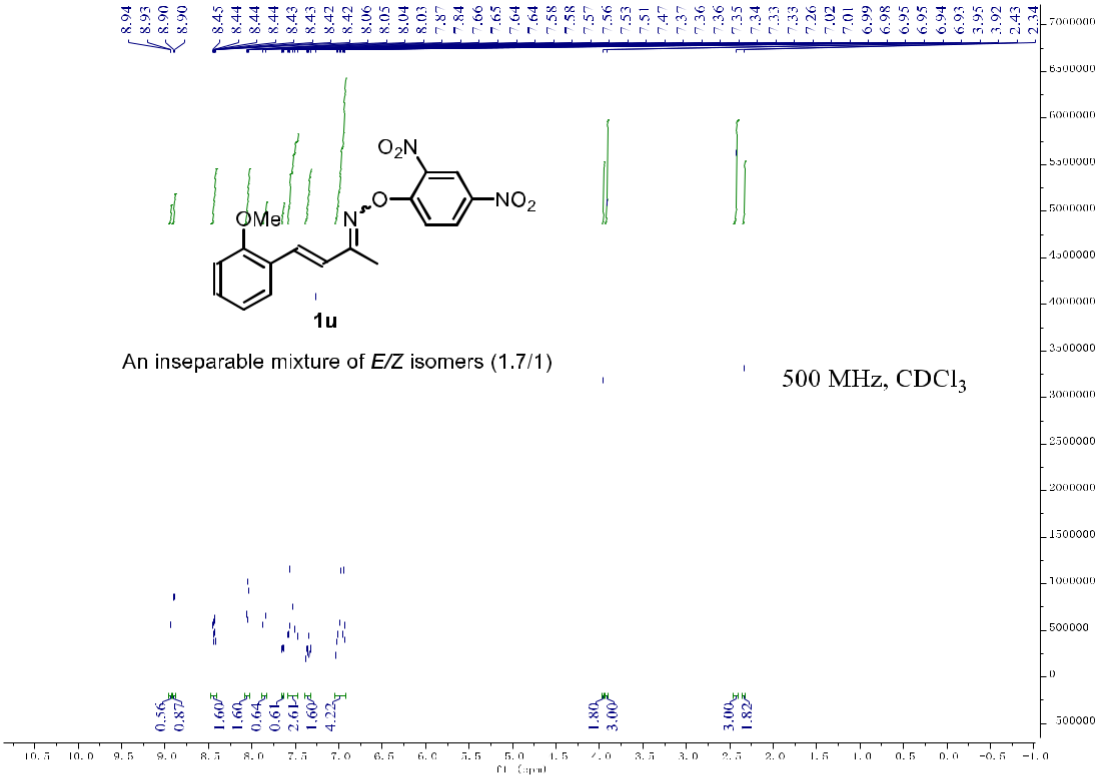


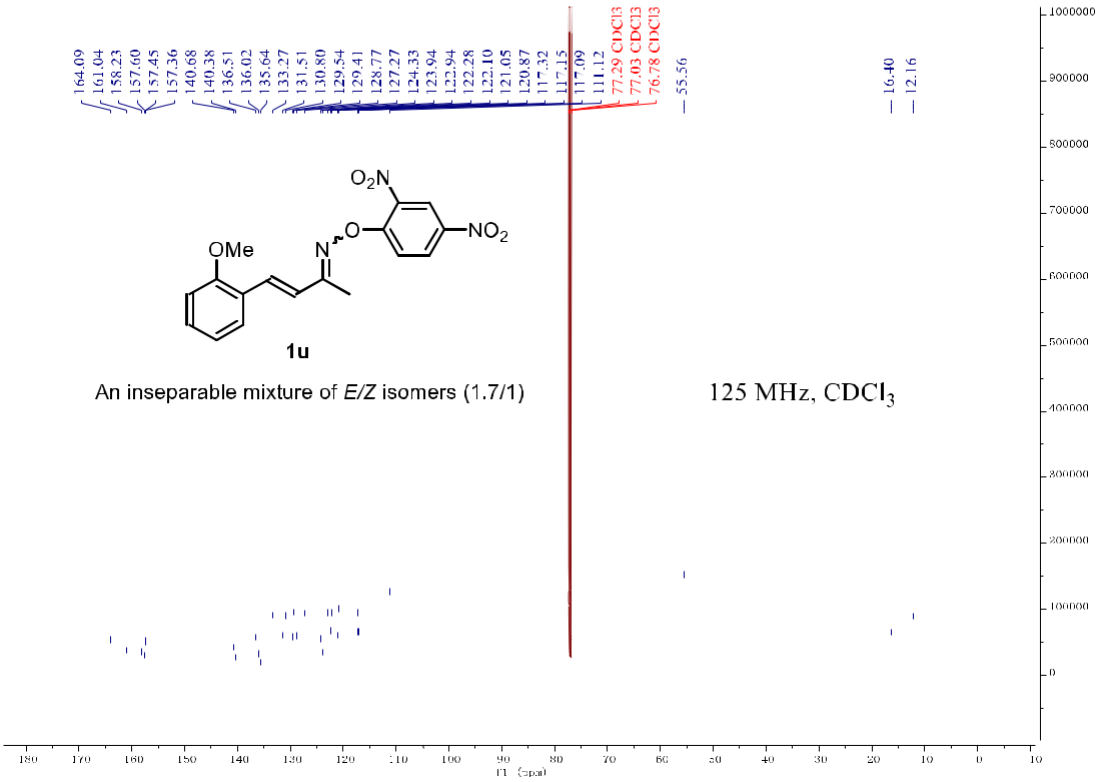
1t

An inseparable mixture of *E/Z* isomers (1.8/1)

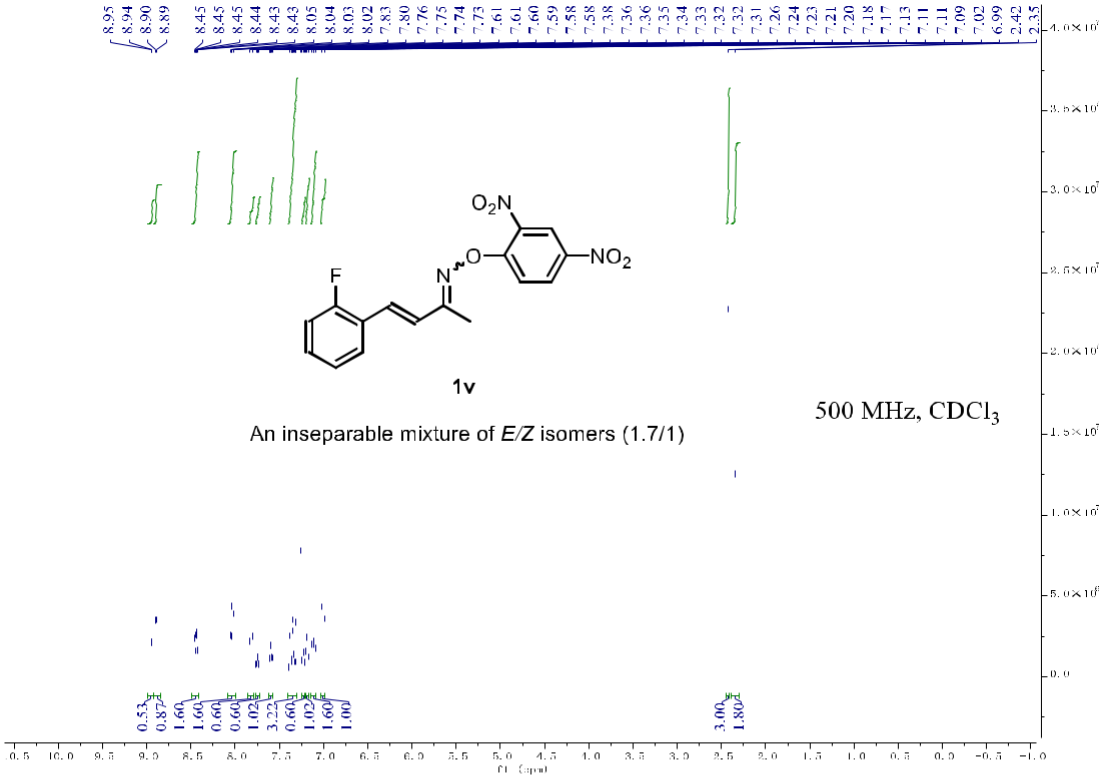
125 MHz, $CDCl_3$

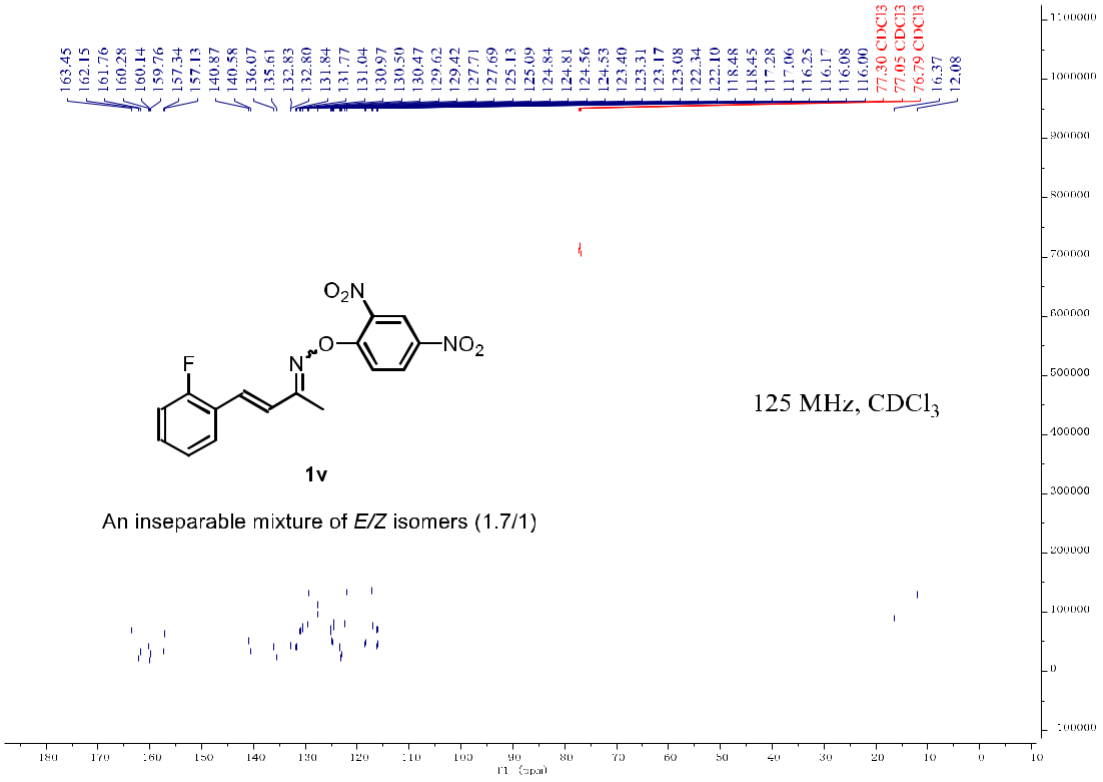
(2E,3E)-4-(2-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1u):



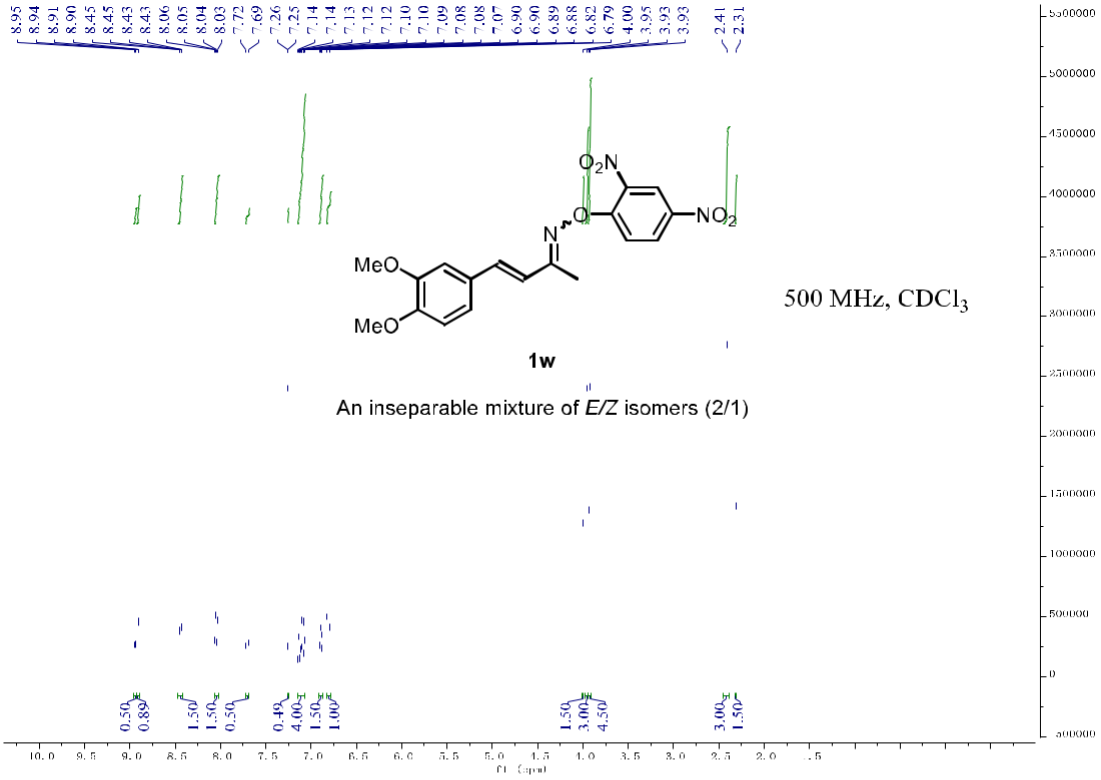


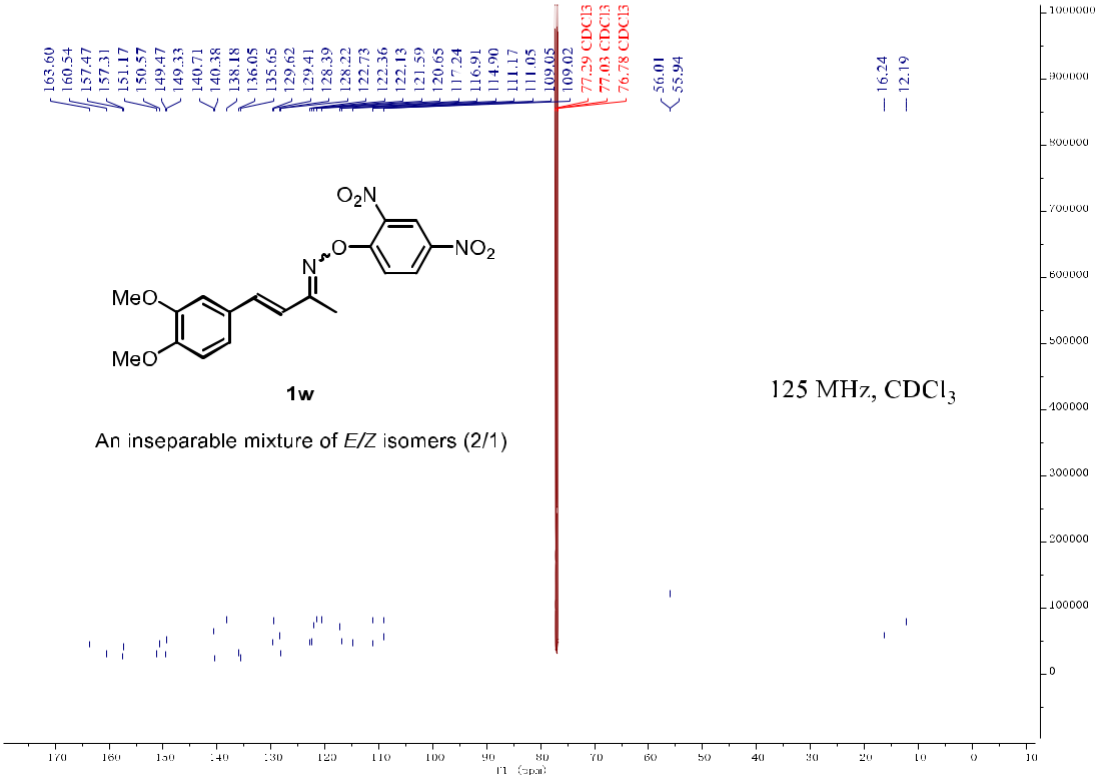
(2E,3E)-4-(2-fluorophenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1v)



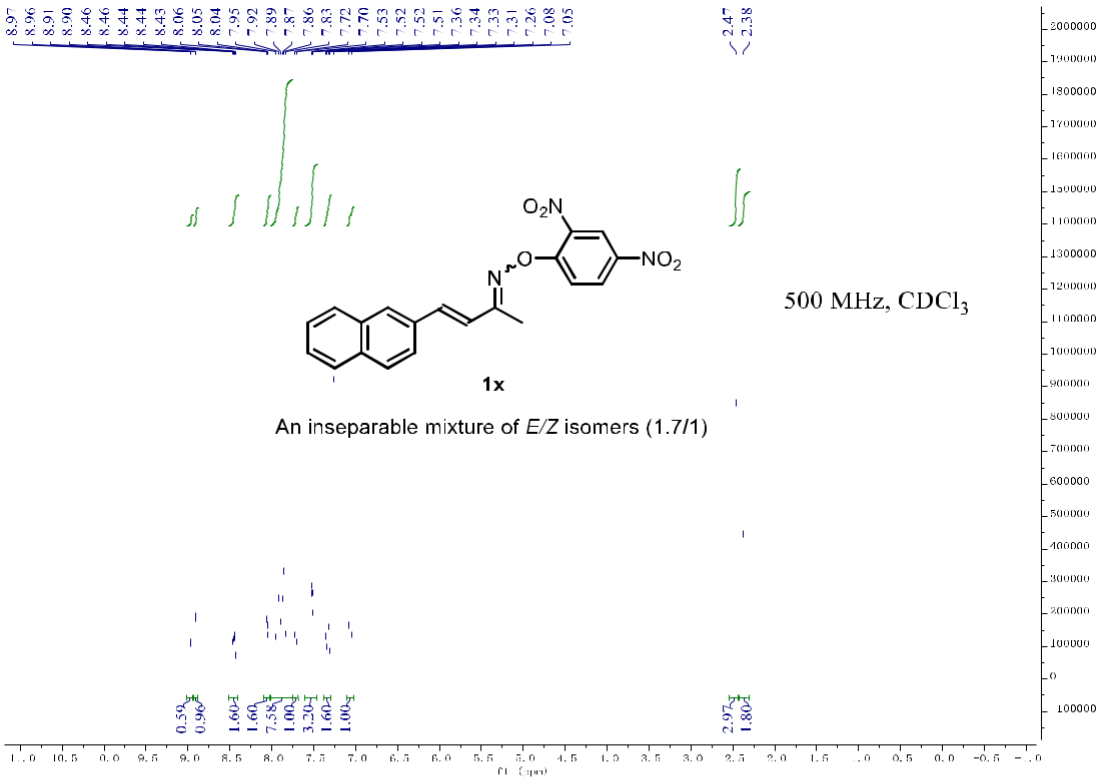


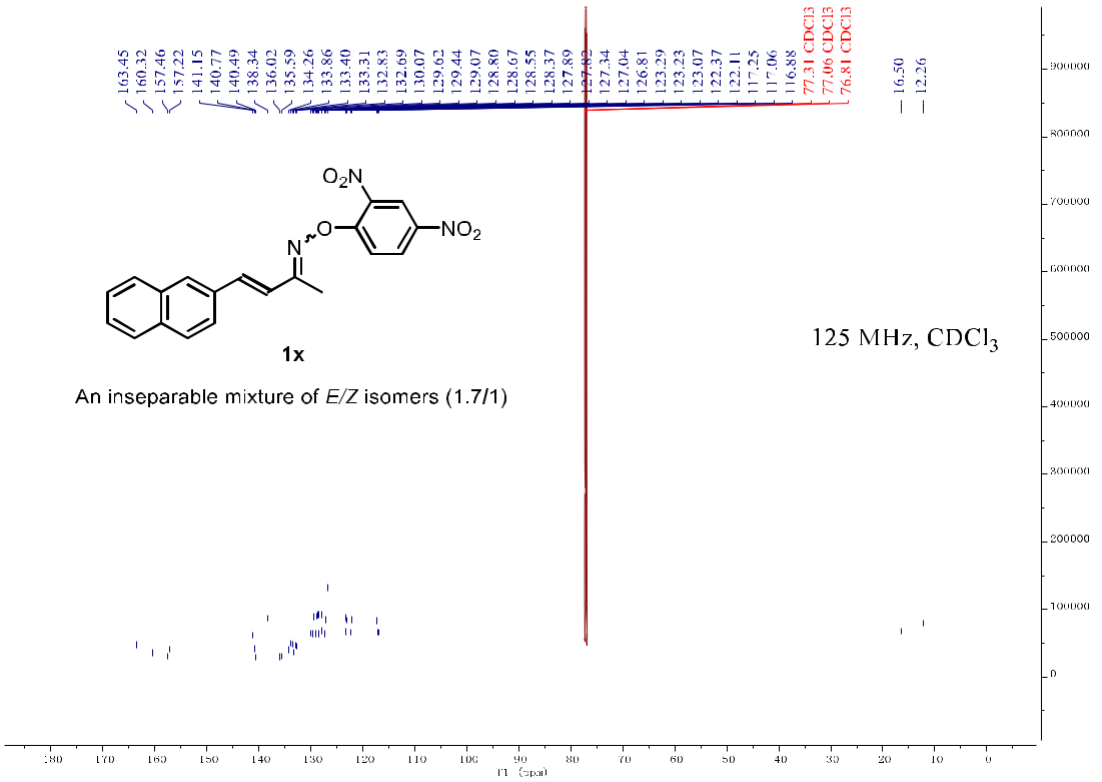
(2E,3E)-4-(3,4-dimethoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1w)



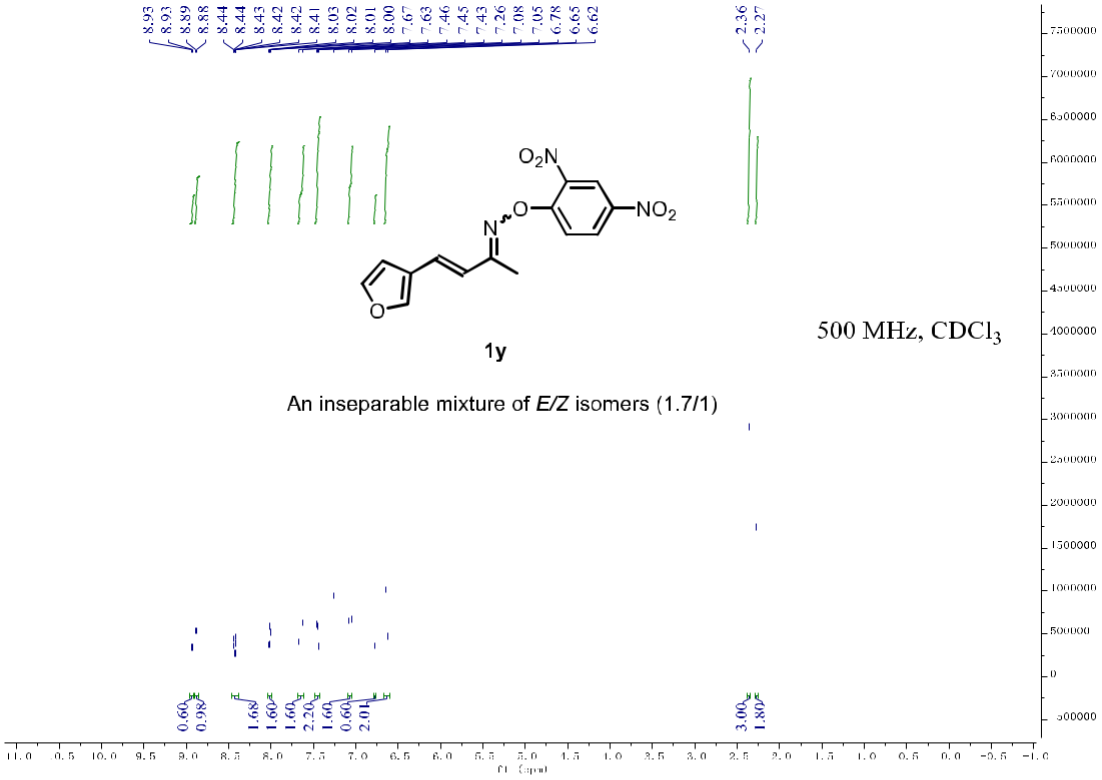


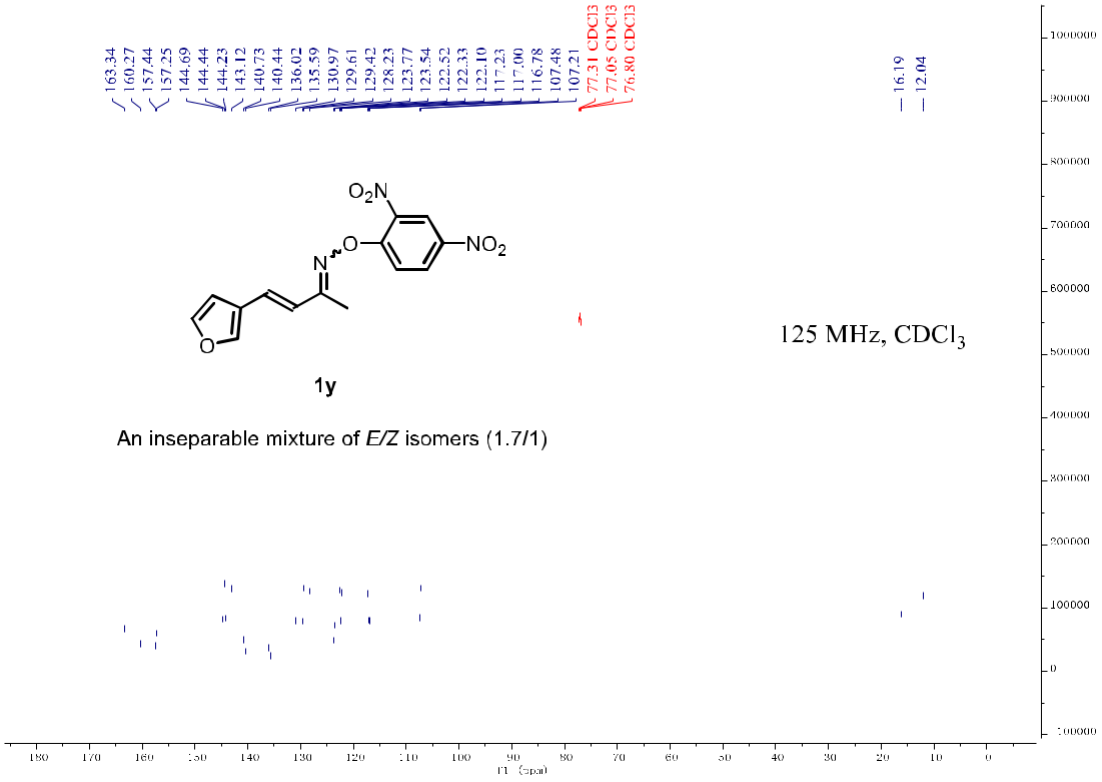
(2E,3E)-4-(naphthalen-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1x)



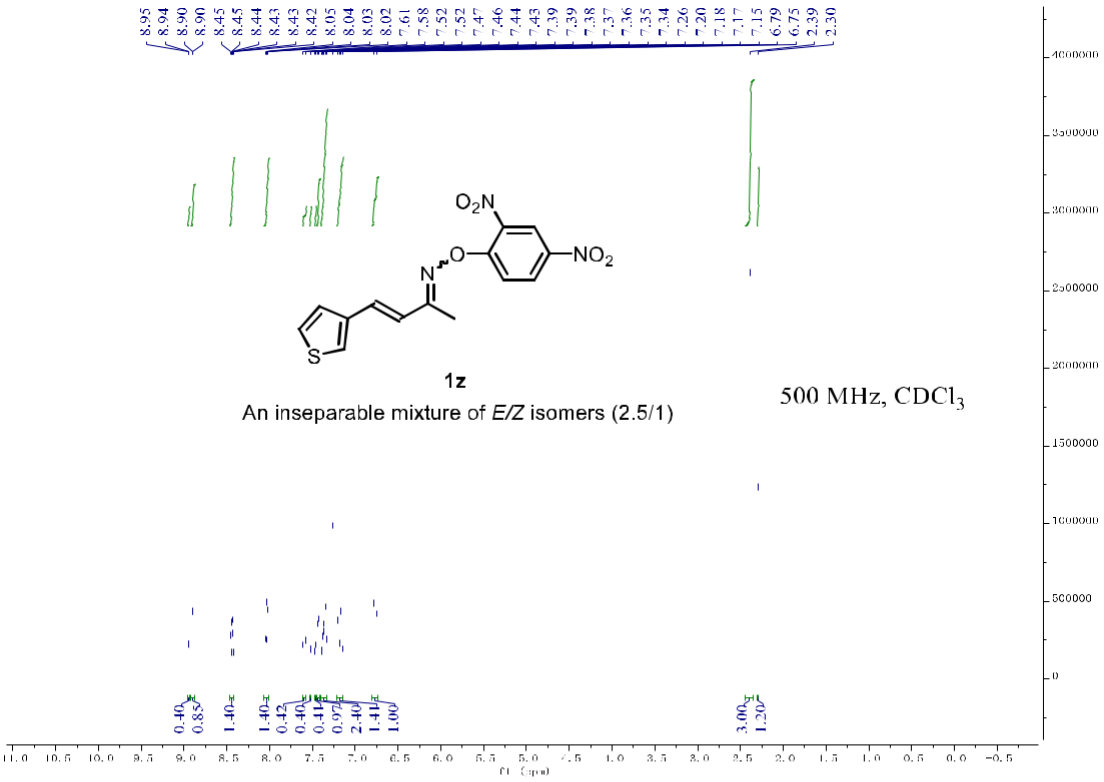


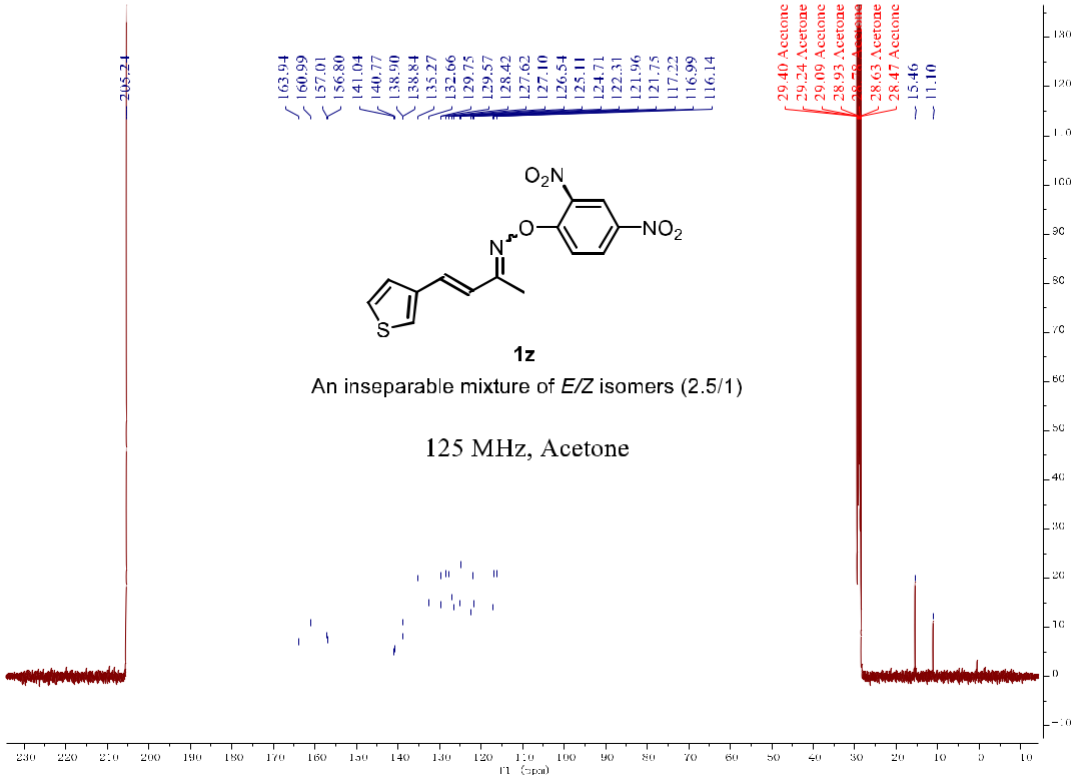
(2E,3E)-N-(2,4-dinitrophenyl)-4-(furan-3-yl)but-3-en-2-imine (1y)



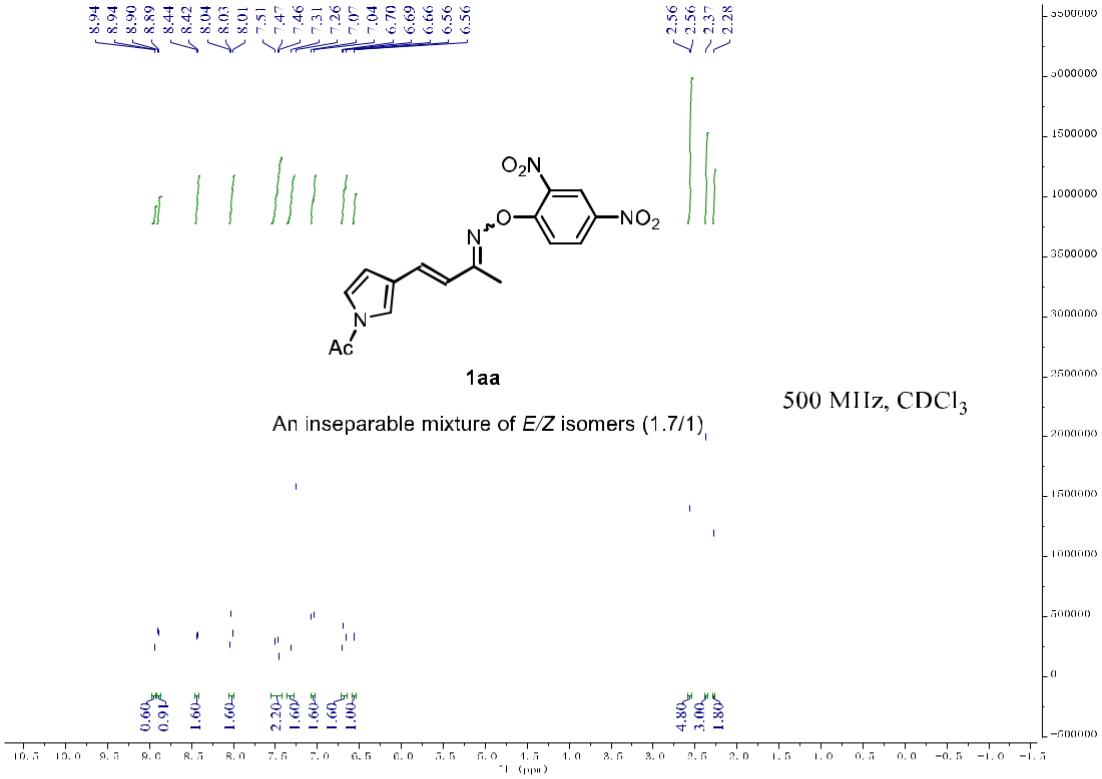


(2E,3E)-N-(2,4-dinitrophenyl)-4-(thiophen-3-yl)but-3-en-2-imine (1z)





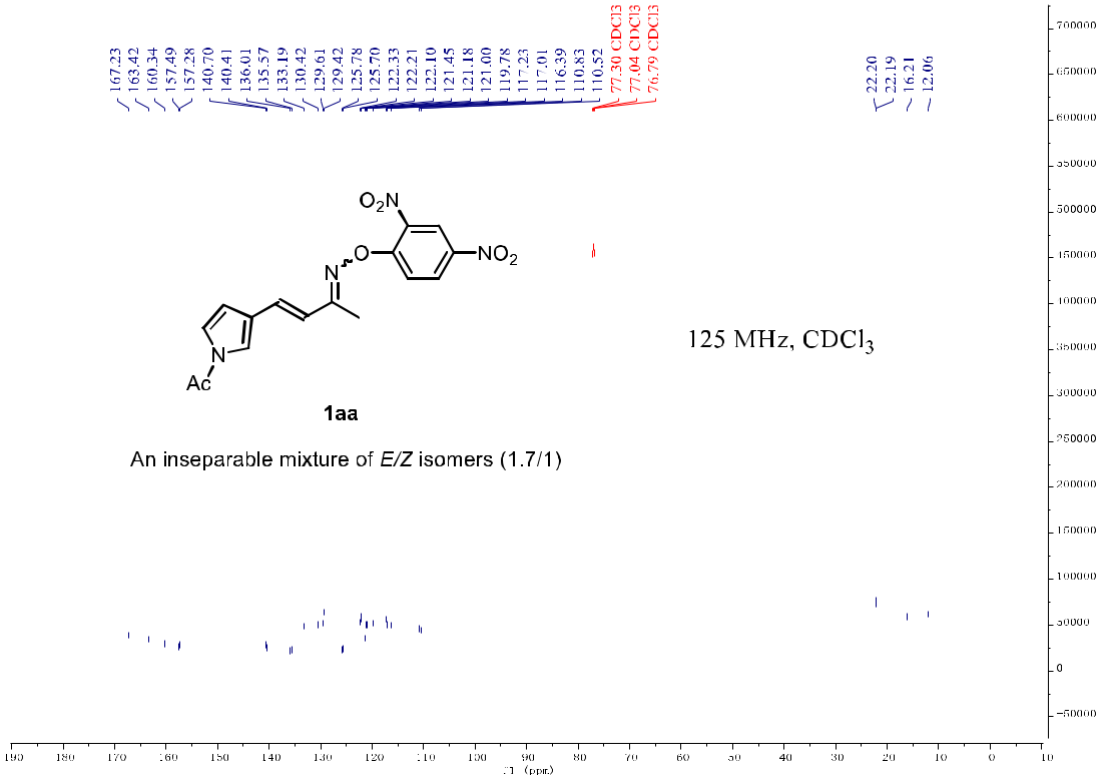
1-(3-((1E,3E)-3-((2,4-dinitrophenyl)imino)but-1-en-1-yl)-1H-pyrrol-1-yl)ethan-1-one (1aa)



1aa

500 MHz, $CDCl_3$

An inseparable mixture of *E/Z* isomers (1.7/1)

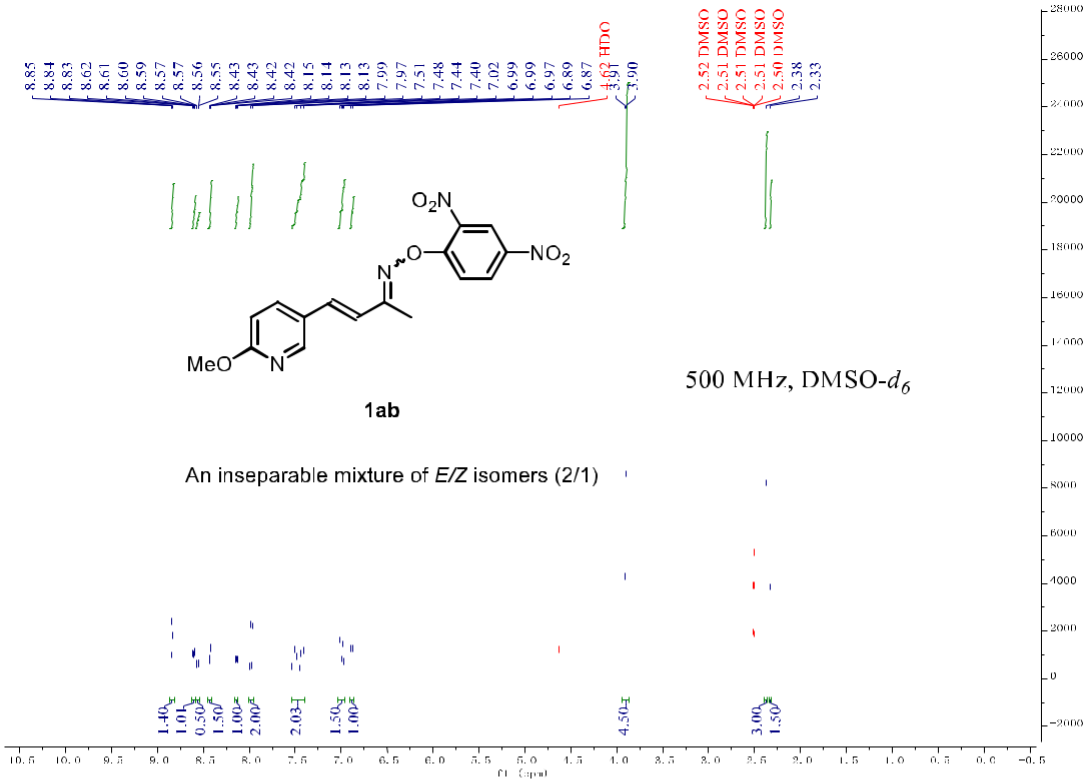


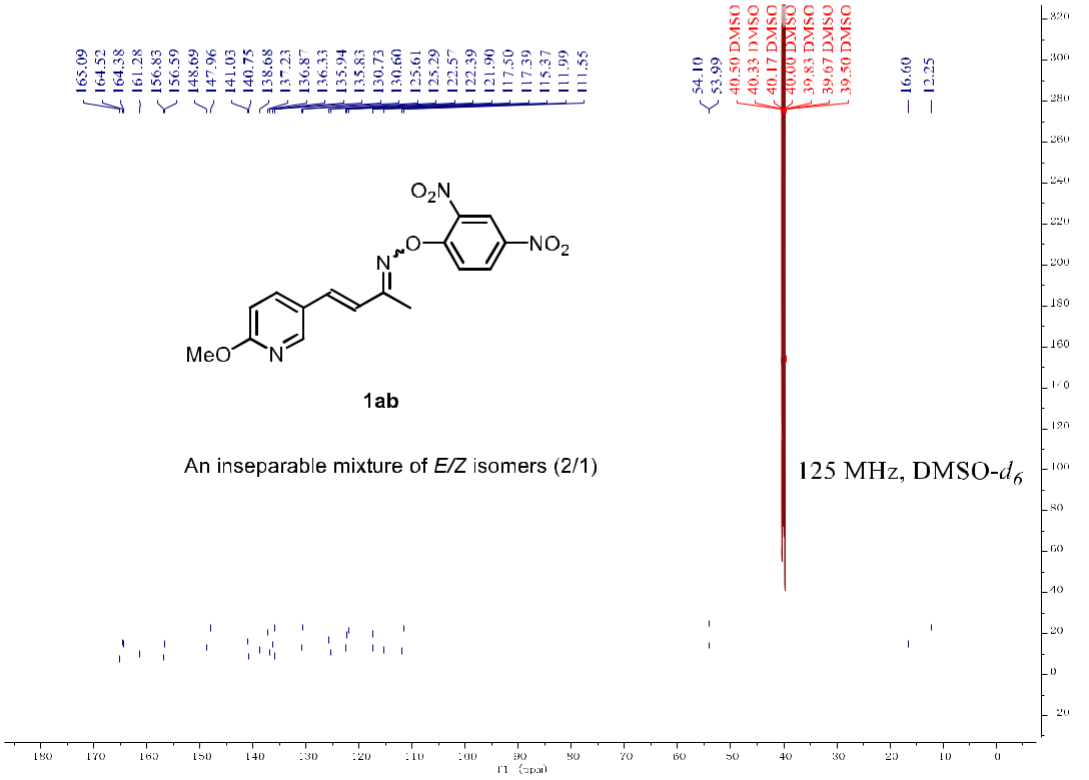
1aa

125 MHz, $CDCl_3$

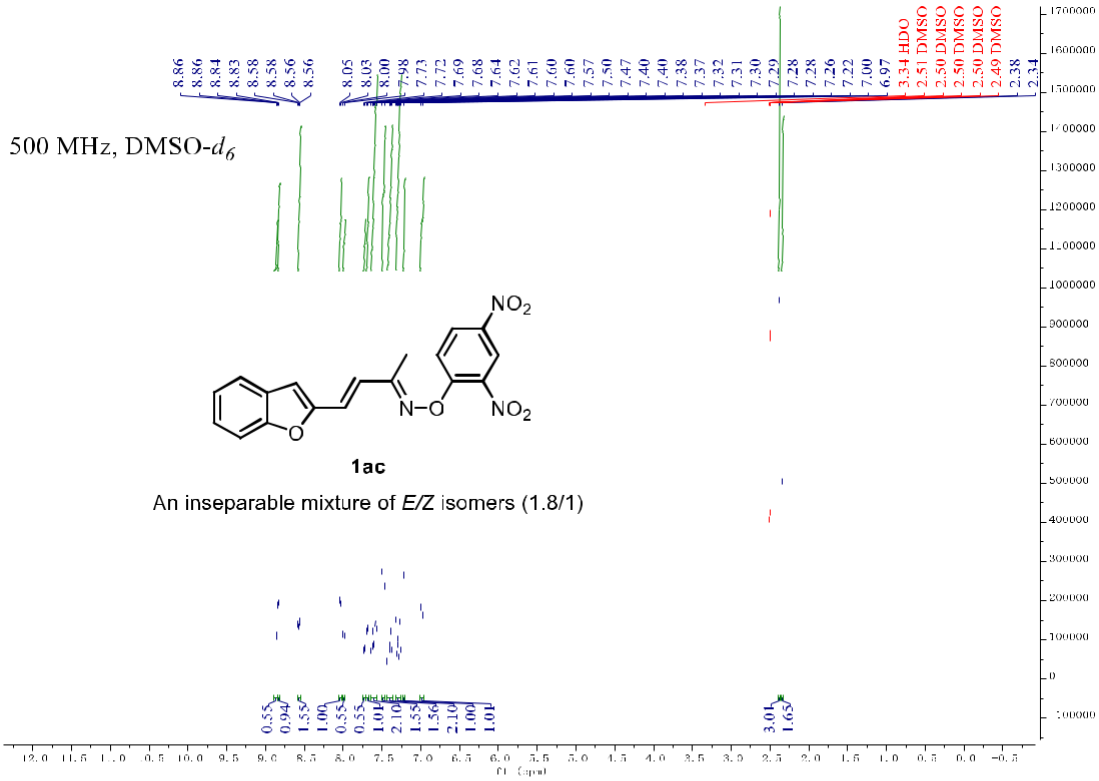
An inseparable mixture of *E/Z* isomers (1.7/1)

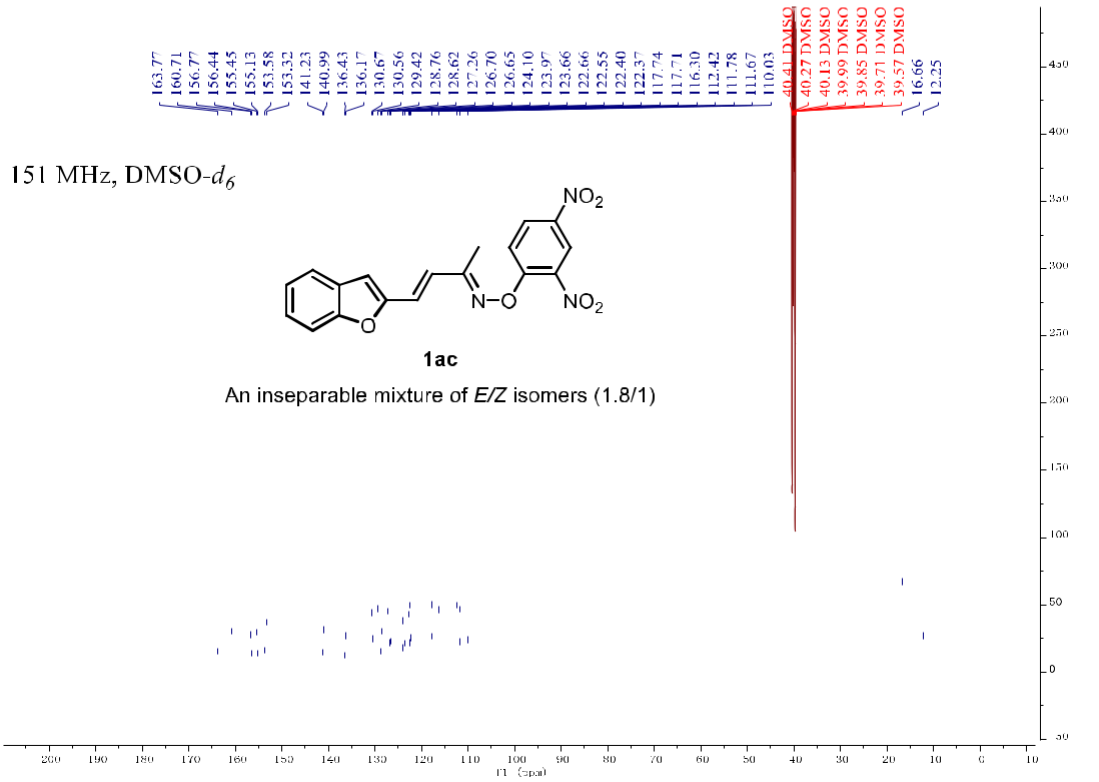
(2E,3E)-4-(6-methoxypyridin-3-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ab)



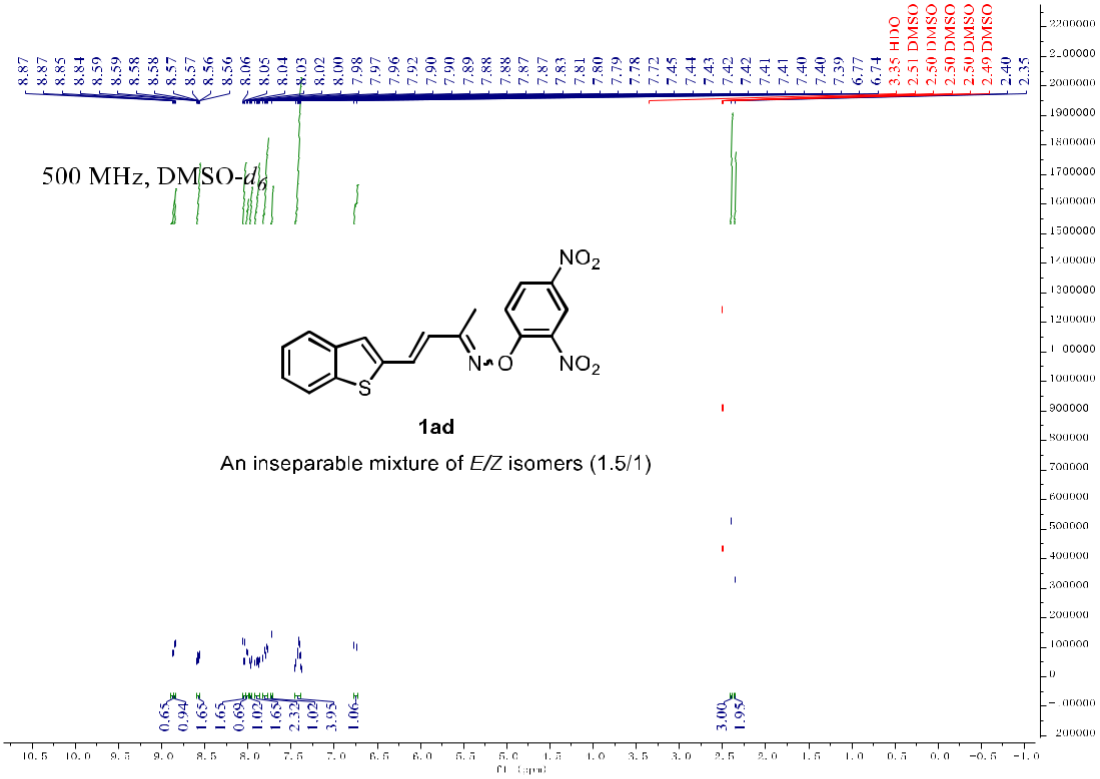


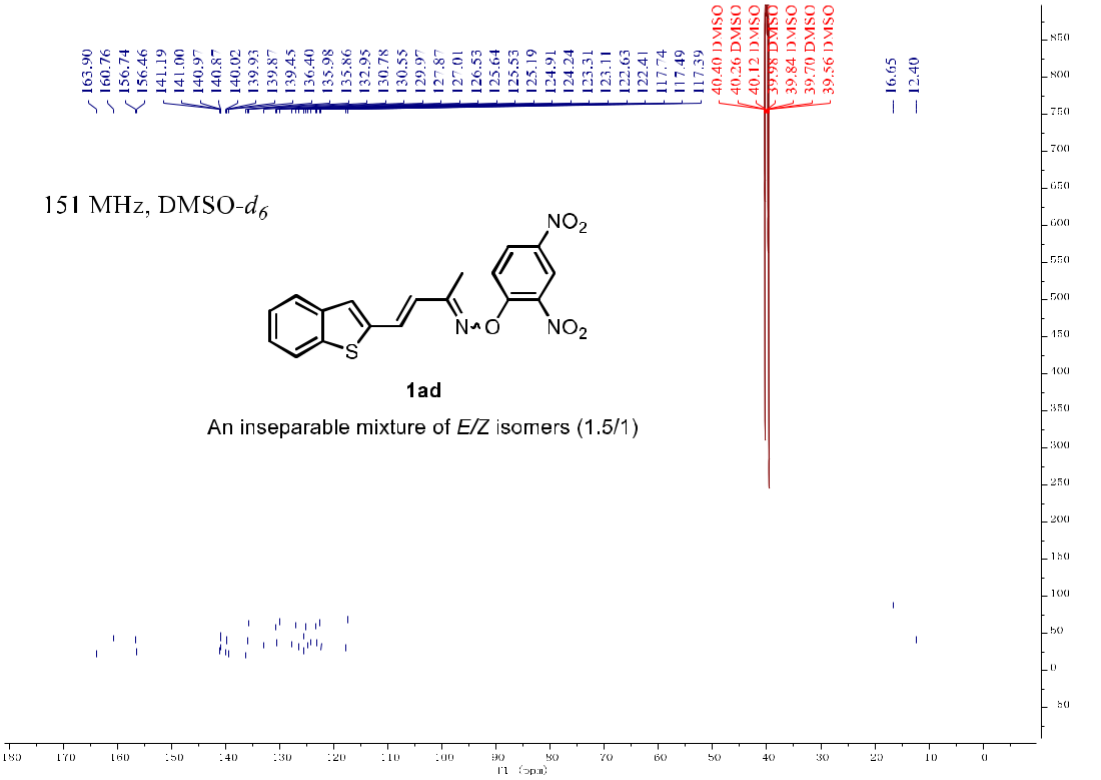
(2E,3E)-4-(benzofuran-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ac)



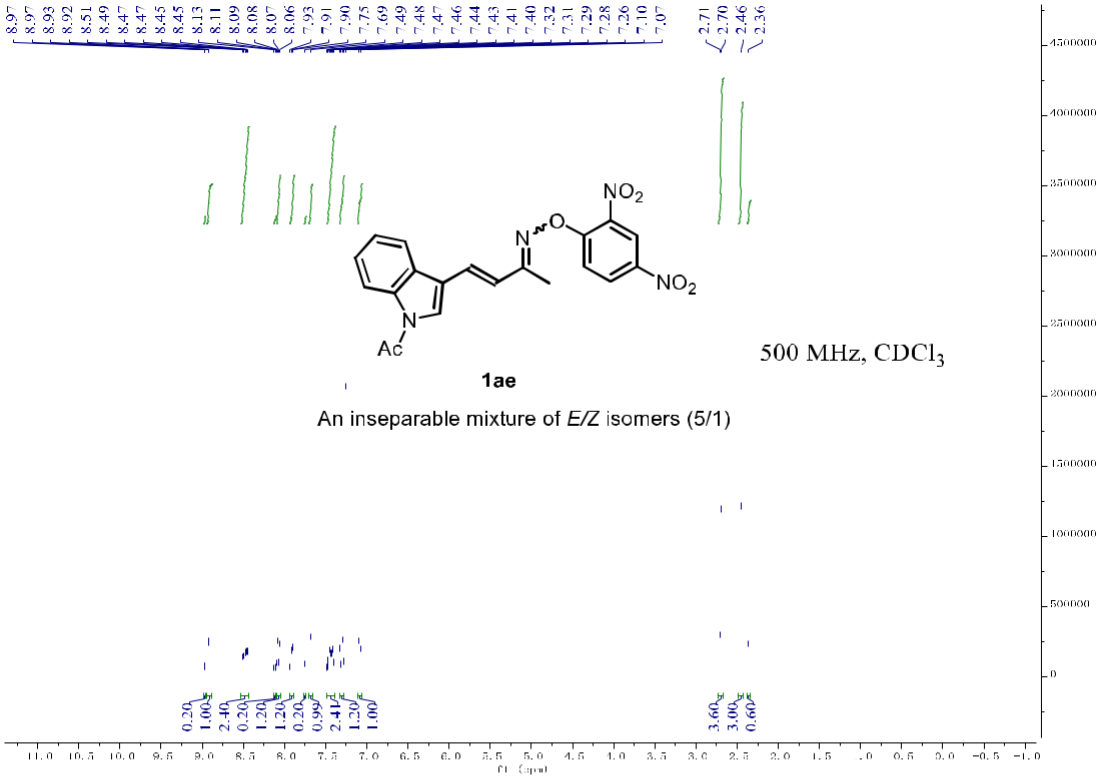


(2E,3E)-4-(benzo[b]thiophen-2-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ad)





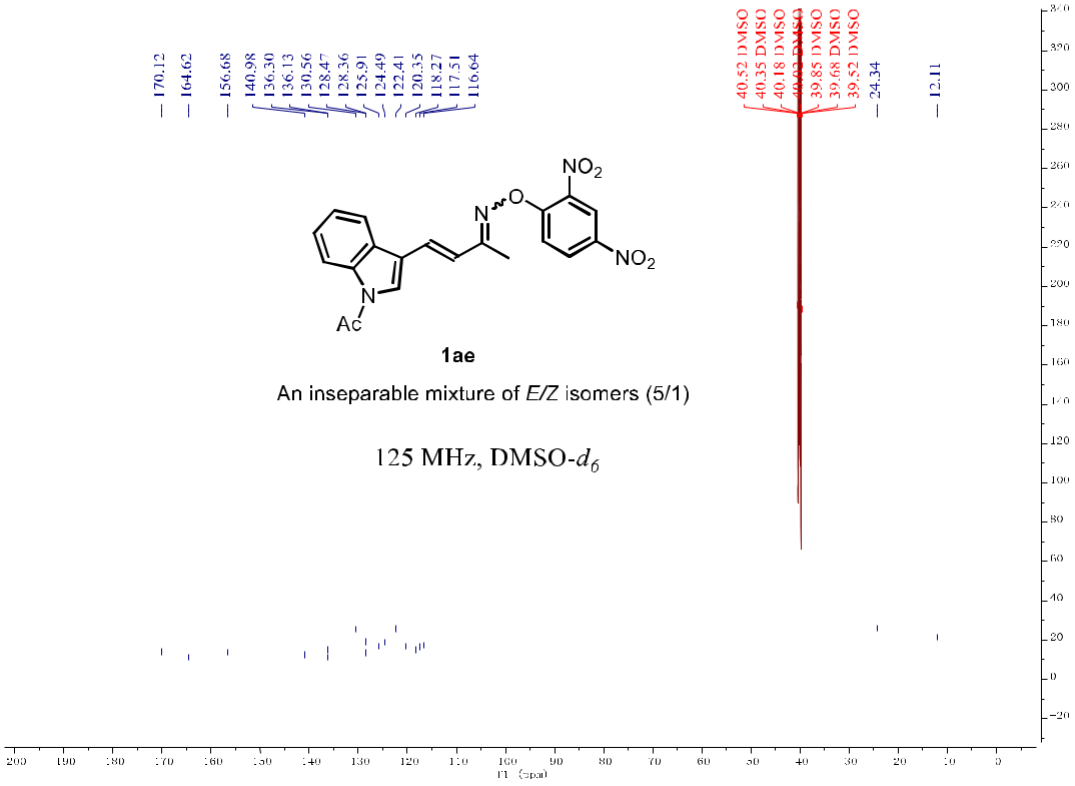
1-(3-((1E,3E)-3-((2,4-dinitrophenyl)imino)but-1-en-1-yl)-1H-indol-1-yl)ethan-1-one (1ae)



500 MHz, $CDCl_3$

1ae

An inseparable mixture of *E/Z* isomers (5/1)

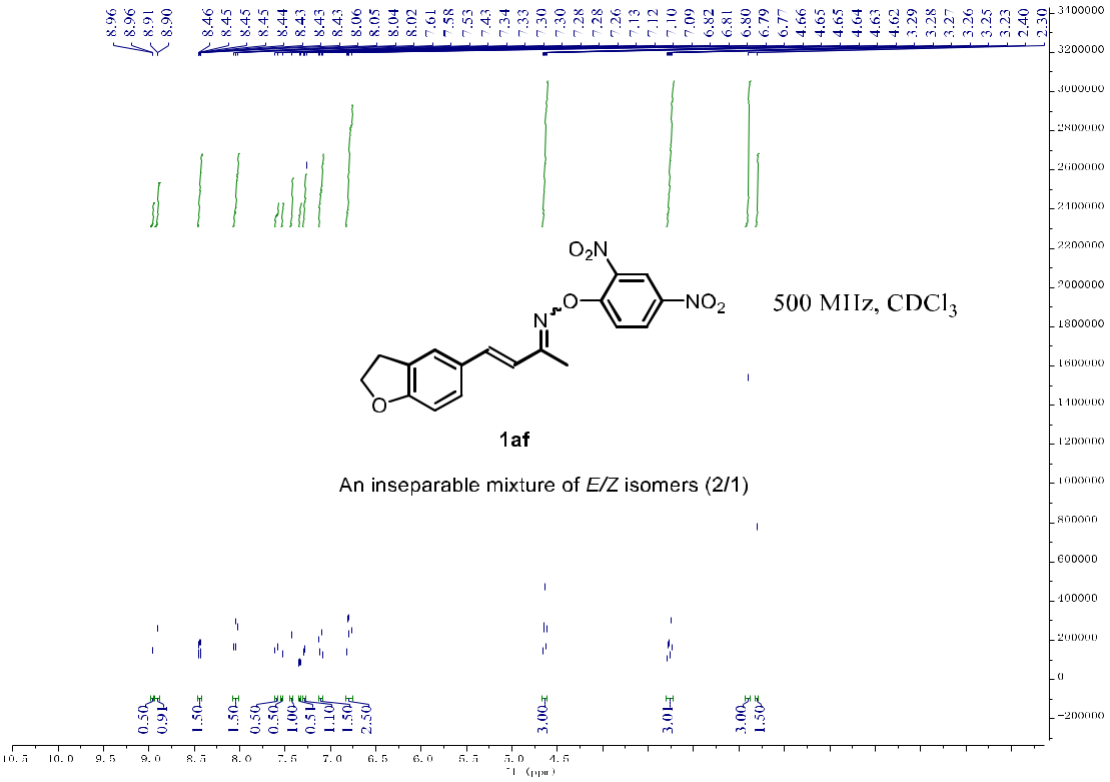


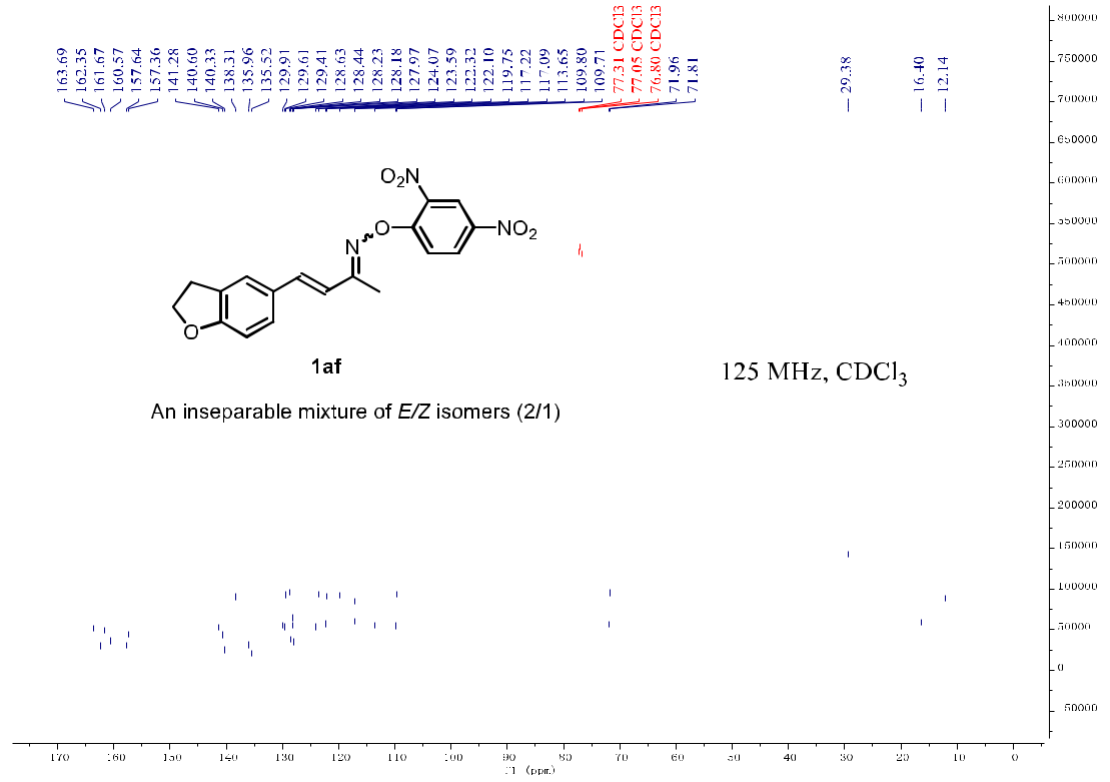
1ae

An inseparable mixture of *E/Z* isomers (5/1)

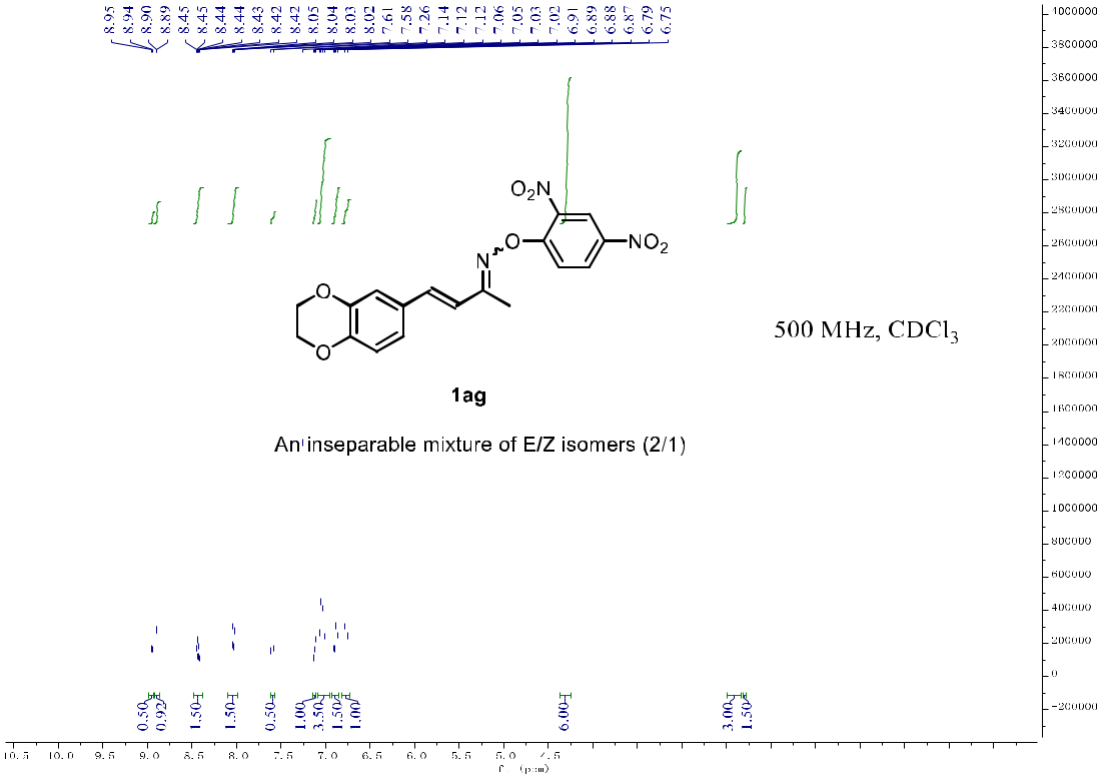
125 MHz, DMSO-d_6

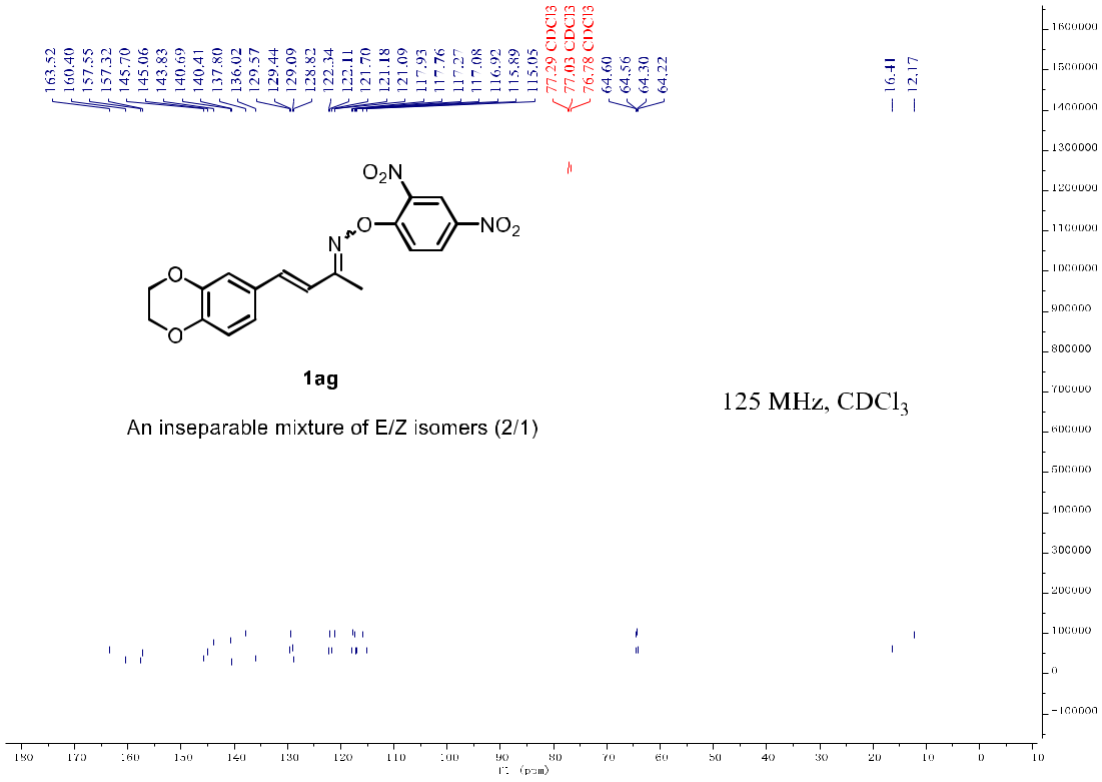
(2E,3E)-4-(2,3-dihydrobenzofuran-5-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1af)



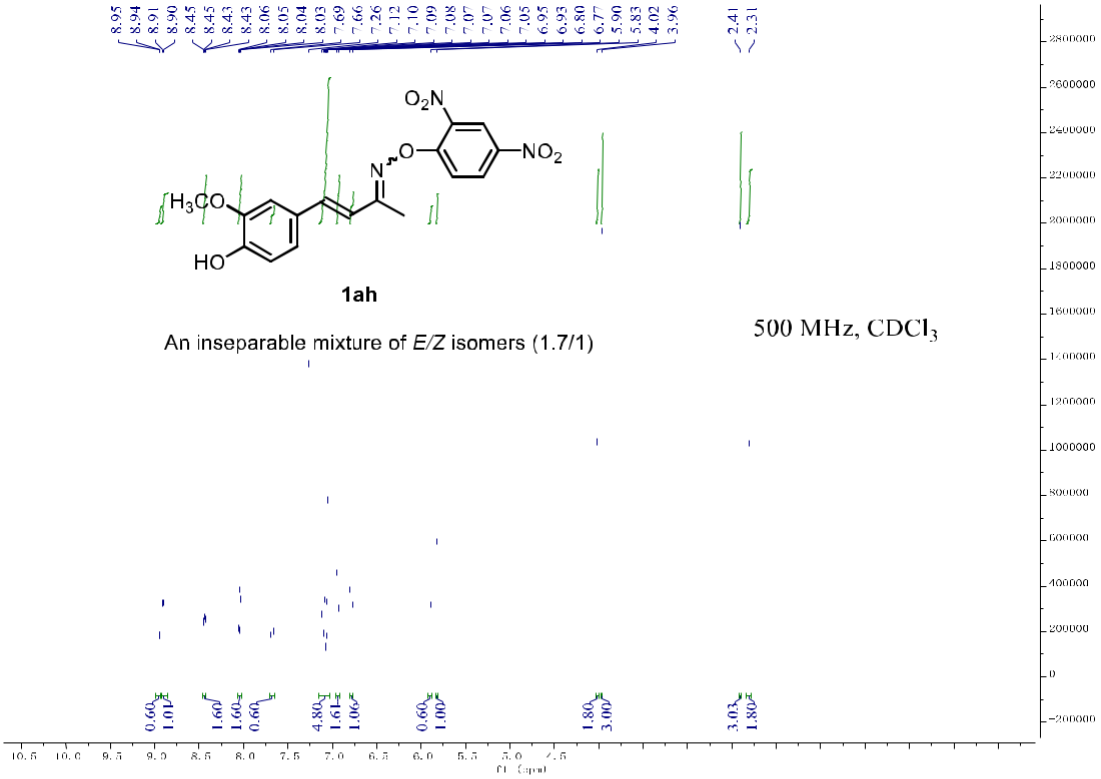


(2E,3E)-4-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ag)





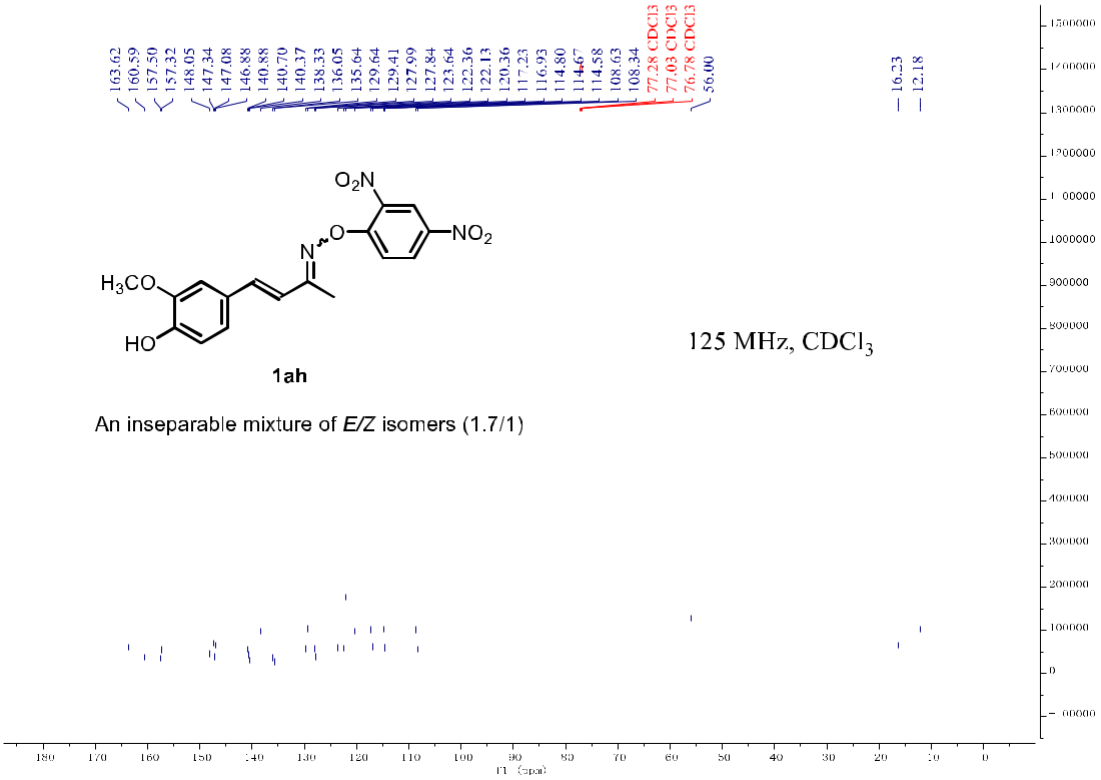
(2E,3E)-4-(4-hydroxy-3-methoxyphenyl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ah)



1ah

An inseparable mixture of *E/Z* isomers (1.7/1)

500 MHz, $CDCl_3$

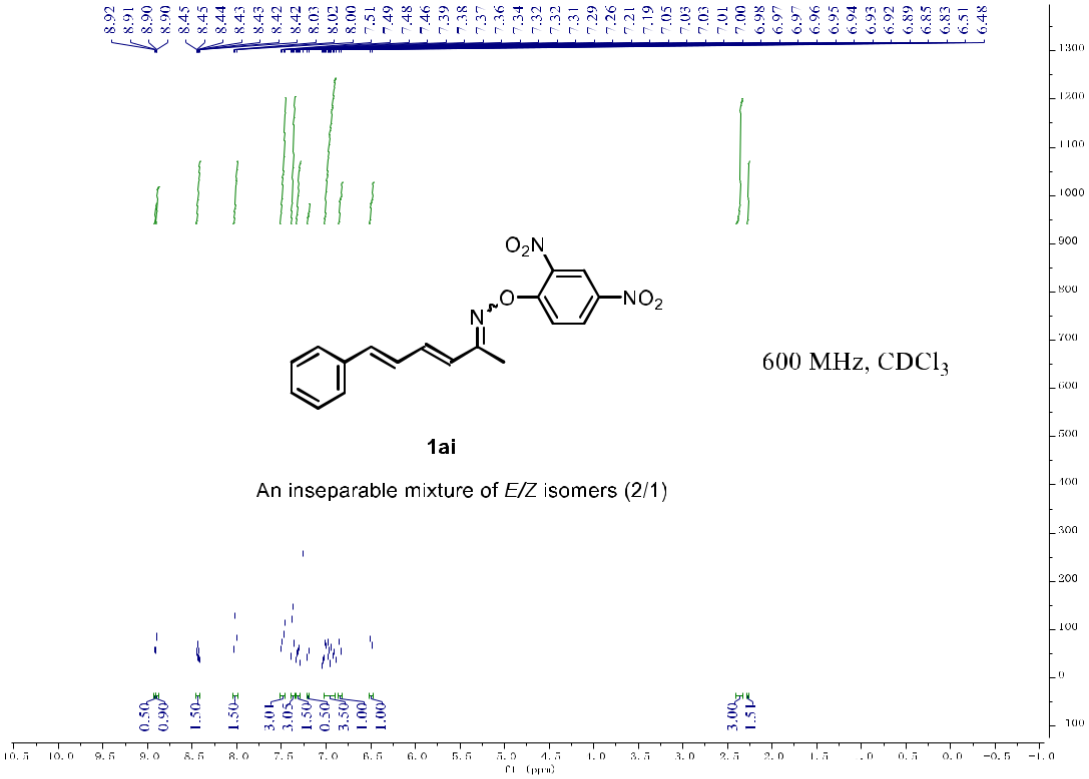


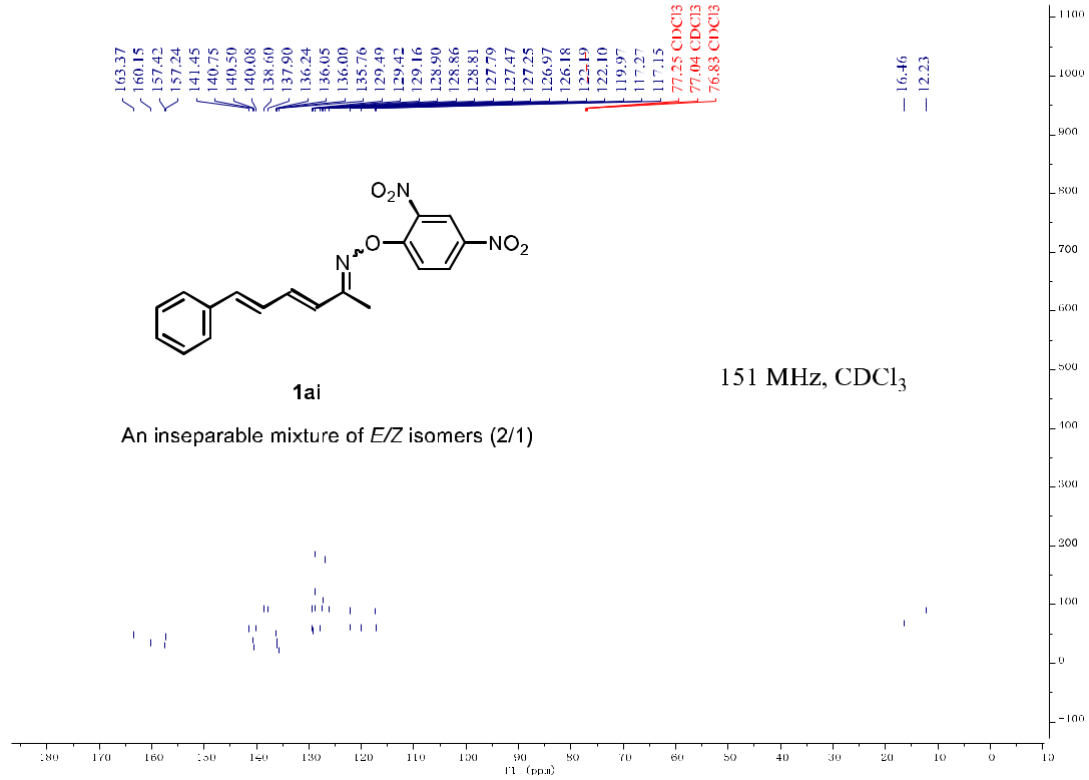
1ah

125 MHz, $CDCl_3$

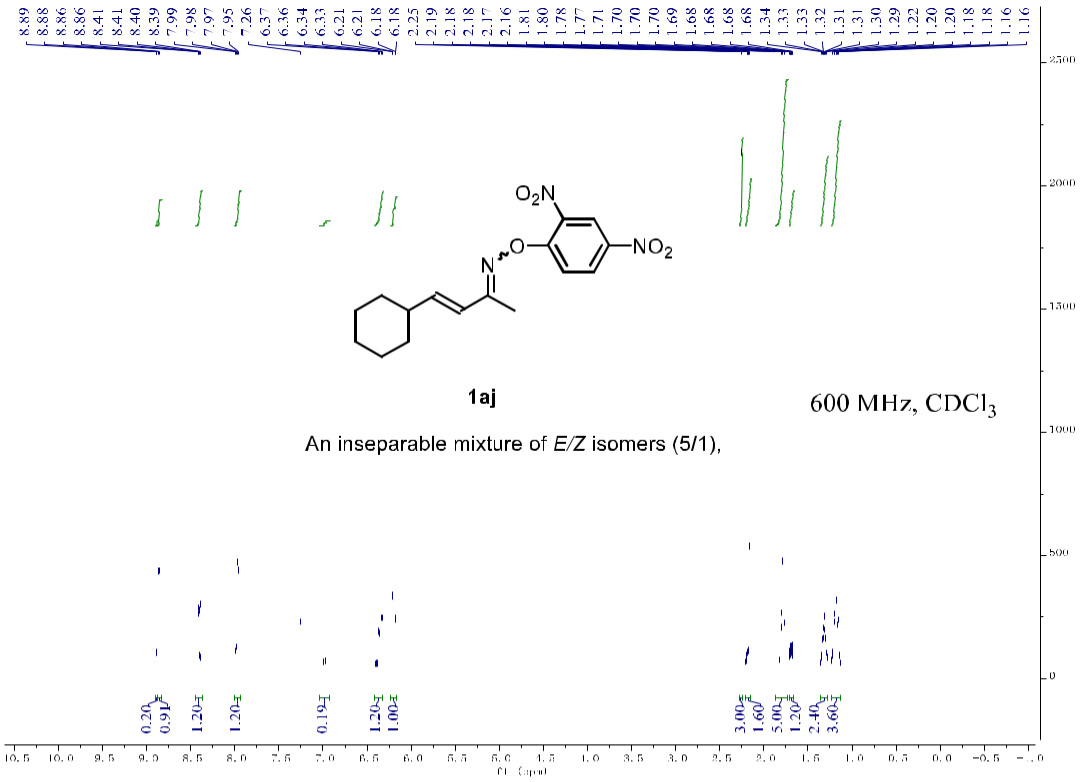
An inseparable mixture of *E/Z* isomers (1.7/1)

(2E,3E,5E)-6-phenylhexa-3,5-dien-2-one O-(2,4-dinitrophenyl) oxime (1ai)





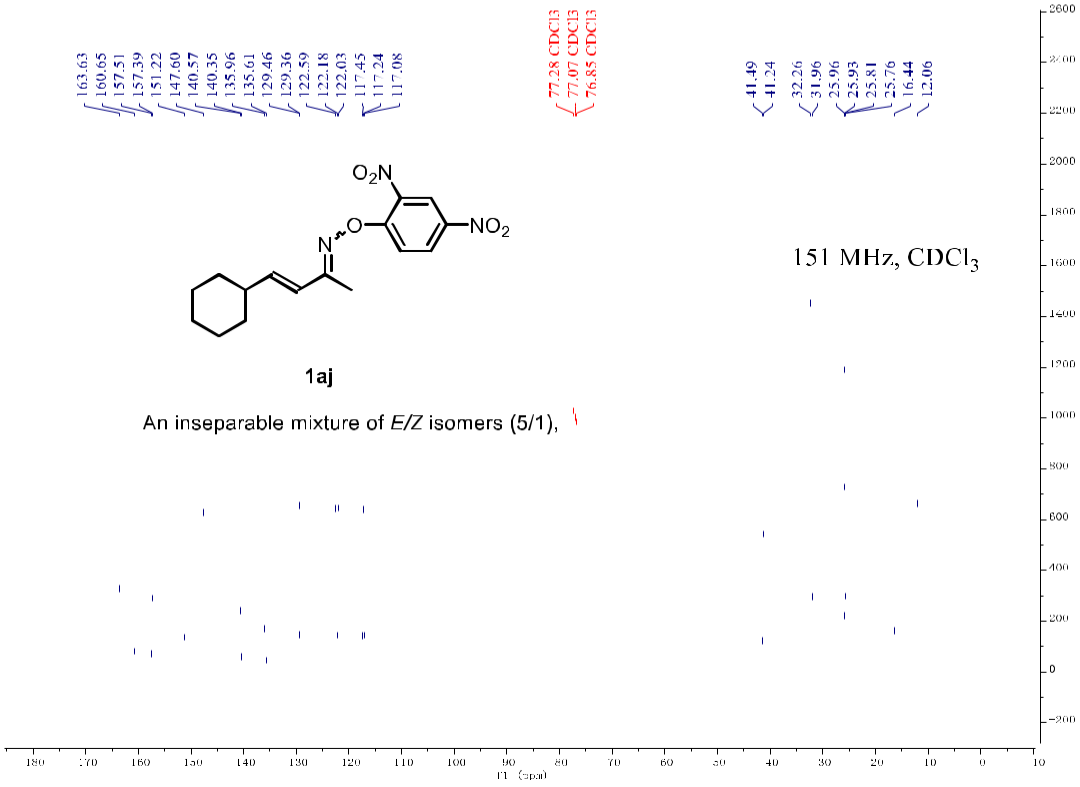
(2E,3E)-4-cyclohexylbut-3-en-2-one O-(2,4-dinitrophenyl) oxime (1aj)



1aj

600 MHz, $CDCl_3$

An inseparable mixture of *E/Z* isomers (5/1),

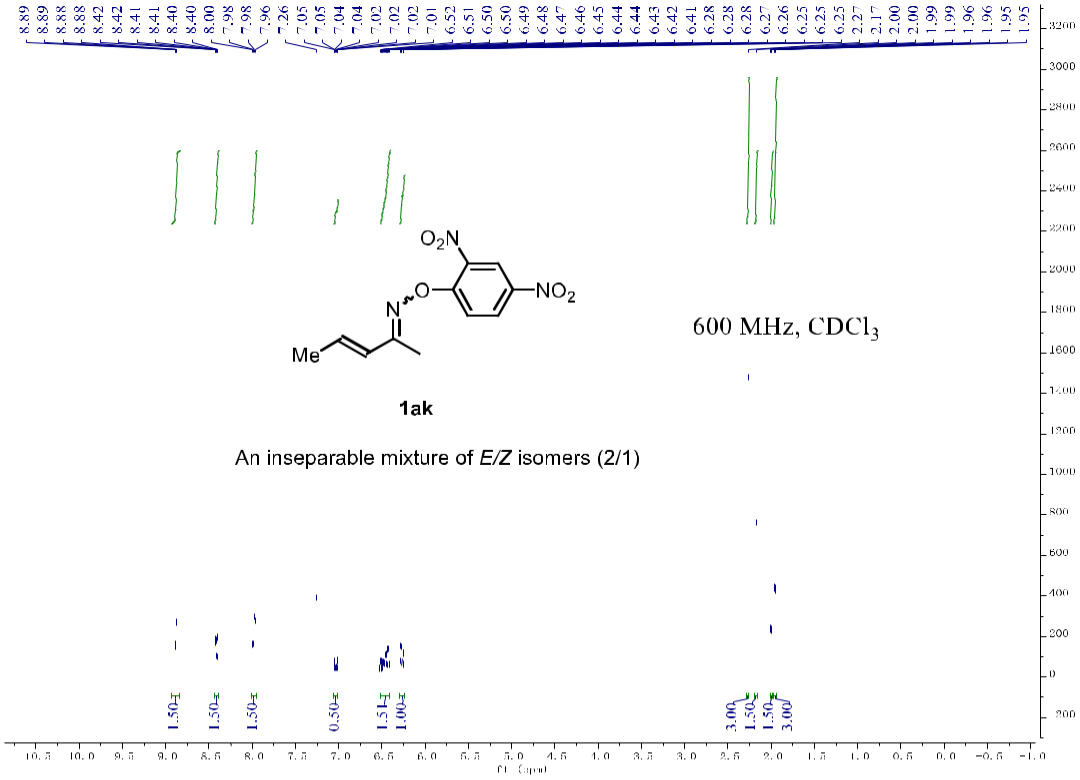


1aj

151 MHz, $CDCl_3$

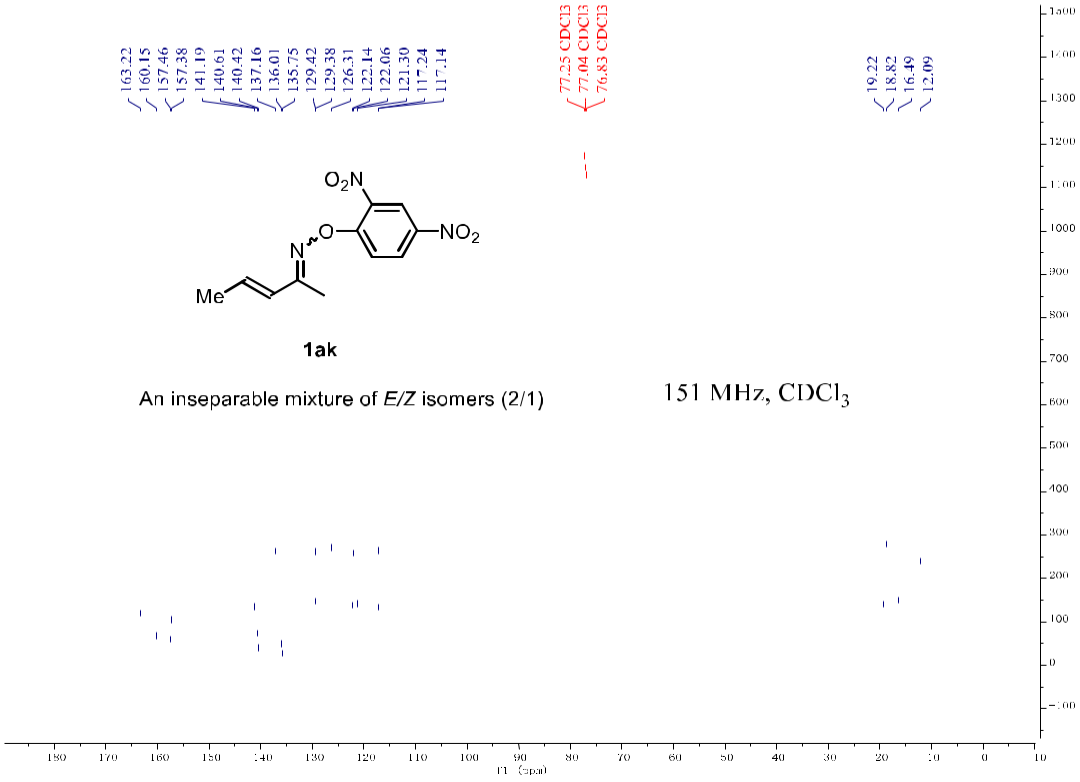
An inseparable mixture of *E/Z* isomers (5/1),

(2E,3E)-pent-3-en-2-one O-(2,4-dinitrophenyl) oxime (1ak)



1ak

An inseparable mixture of *E/Z* isomers (2/1)

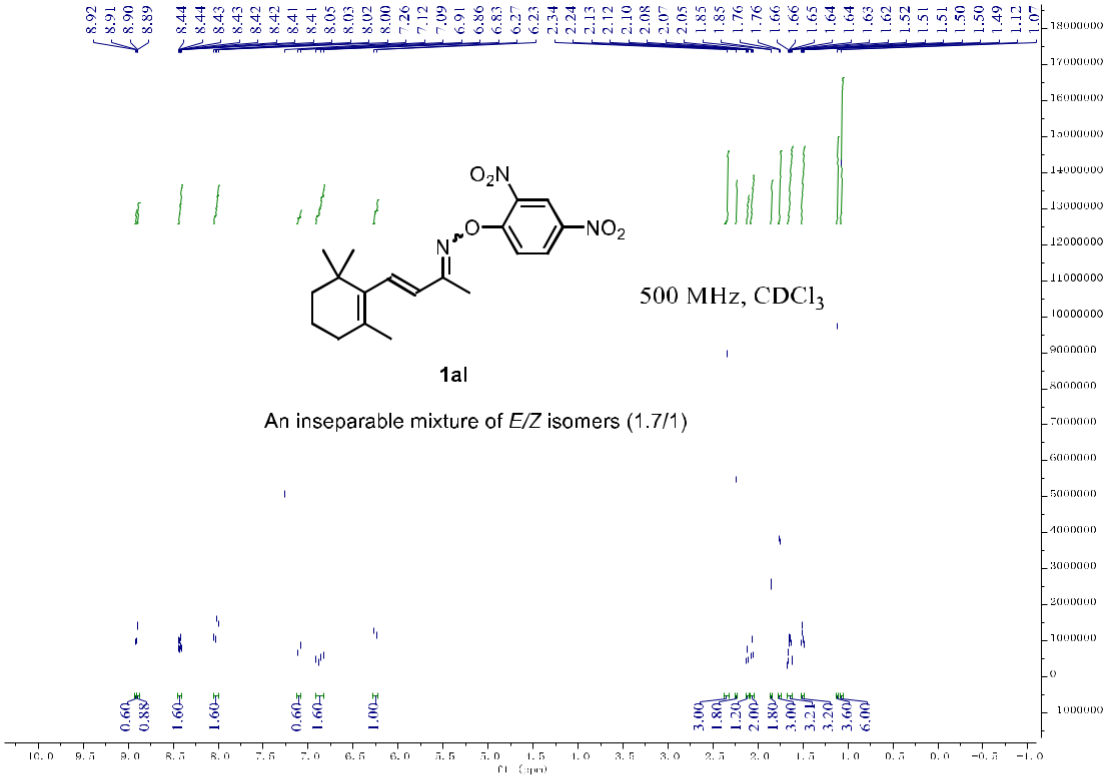


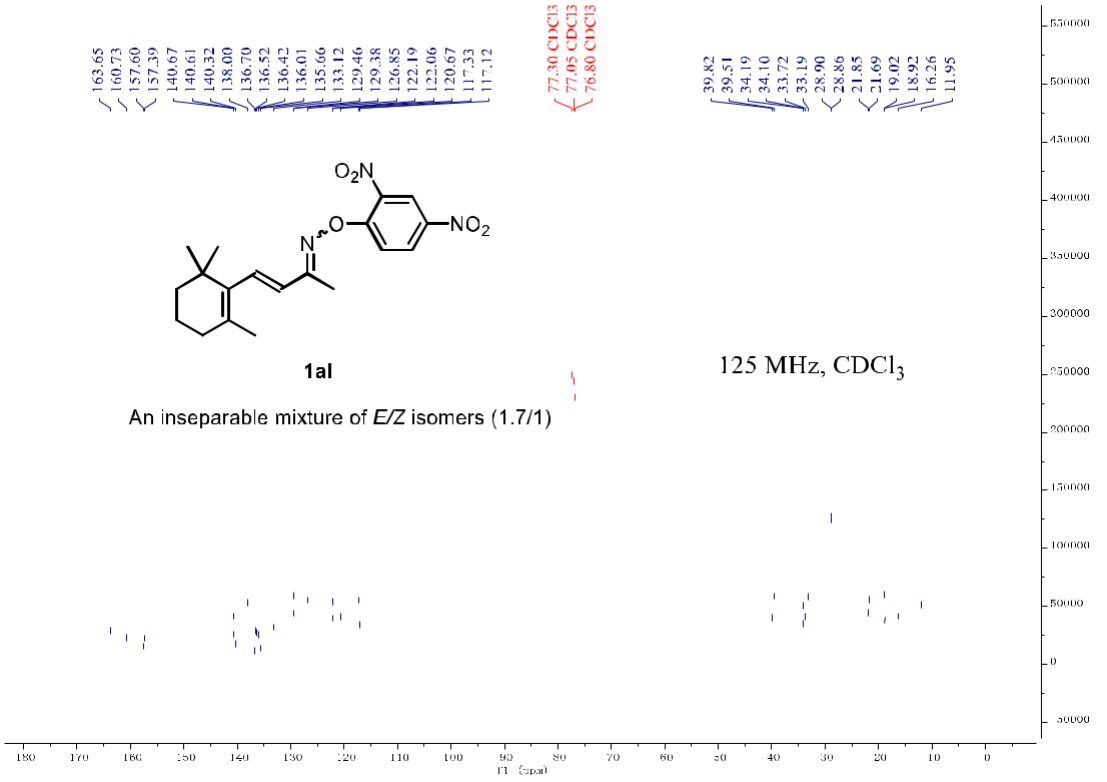
1ak

An inseparable mixture of *E/Z* isomers (2/1)

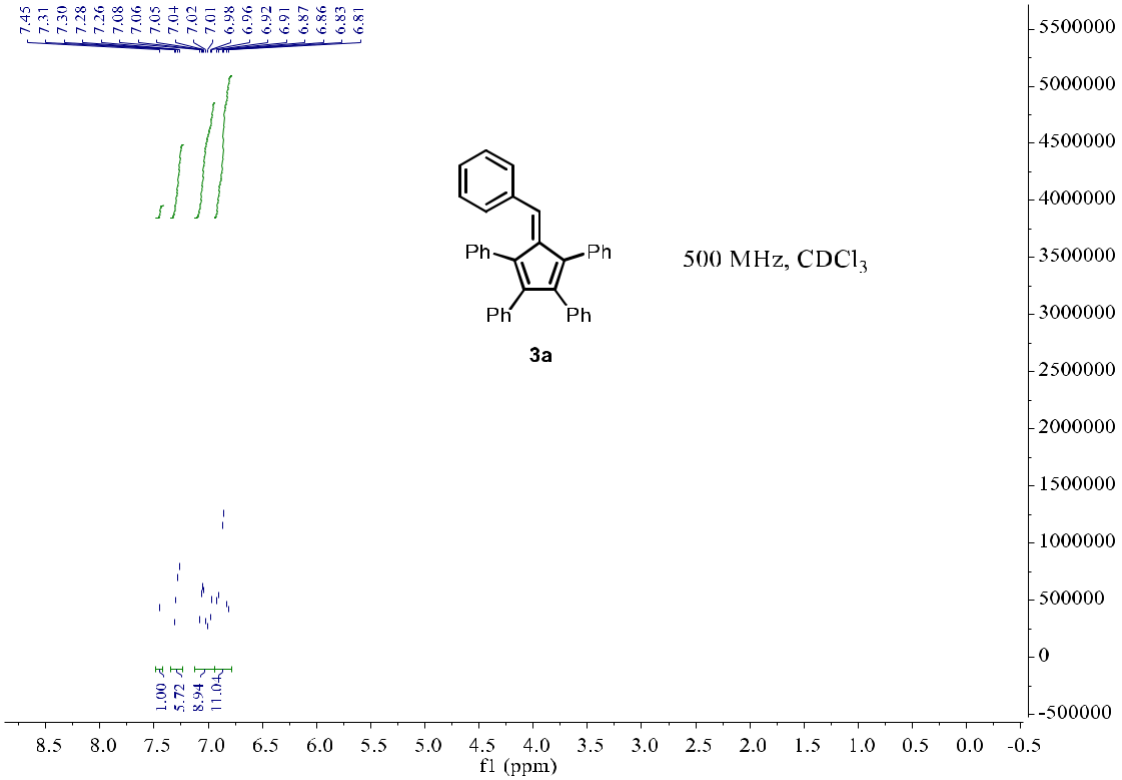
151 MHz, $CDCl_3$

(2E,3E)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-one O-(2,4-dinitrophenyl) oxime (1al)

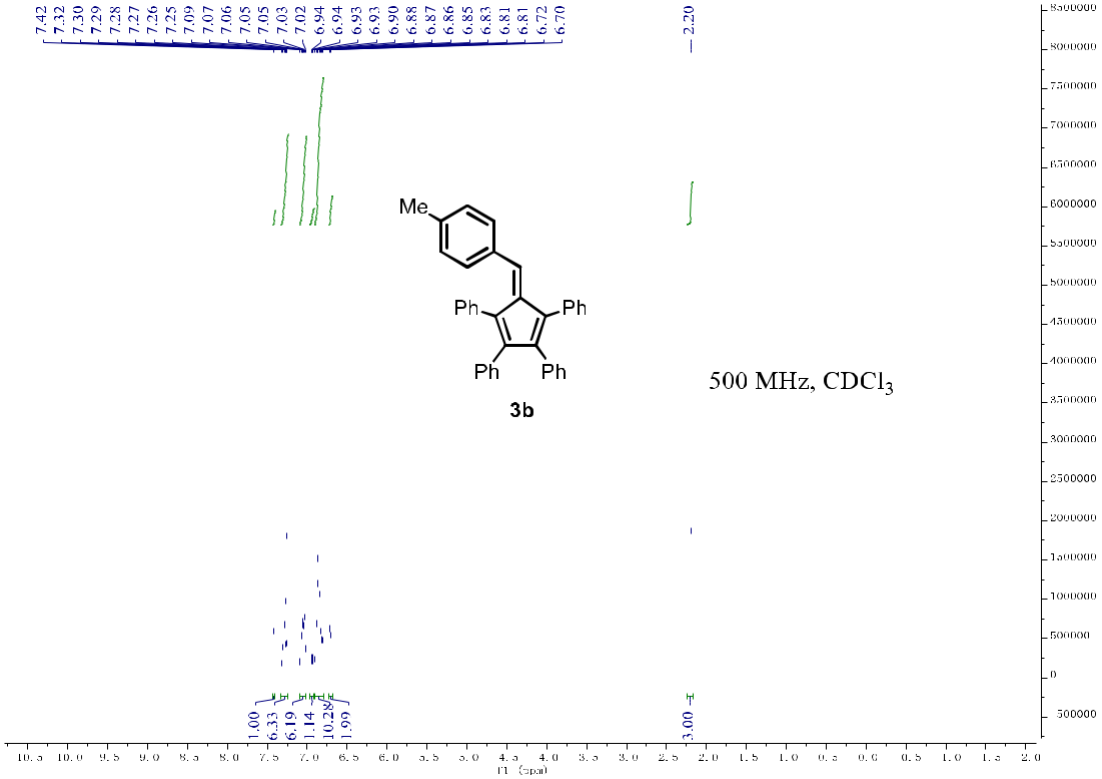




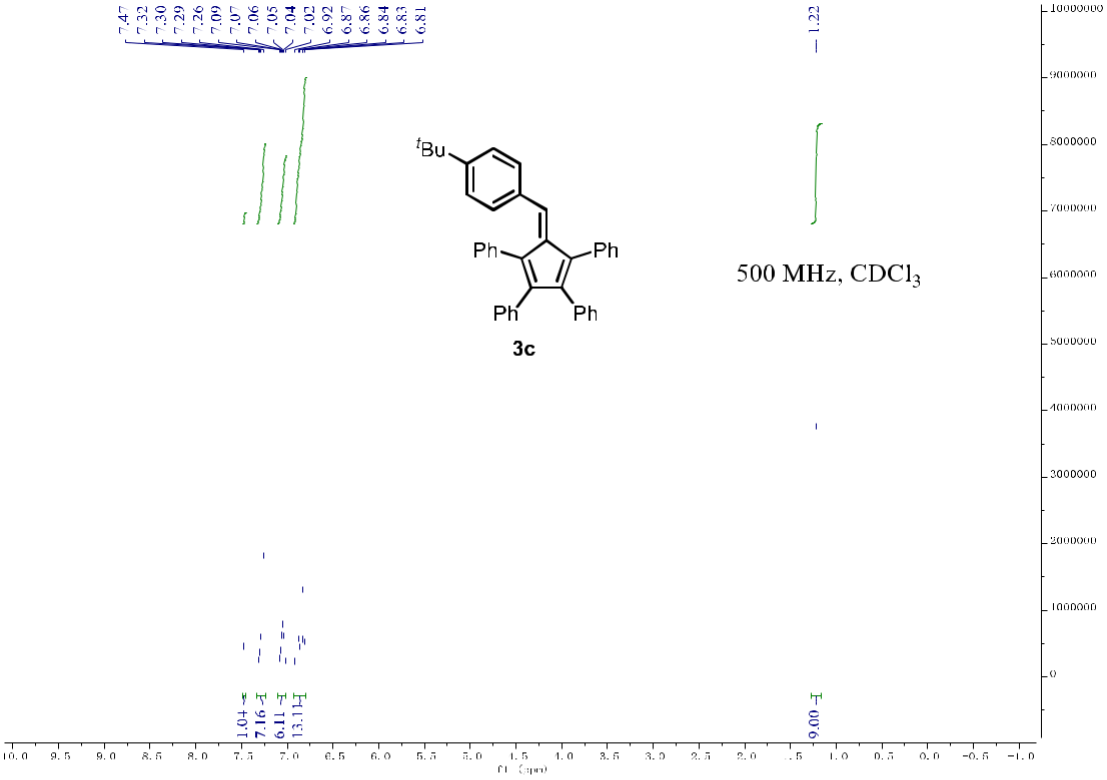
(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3a)

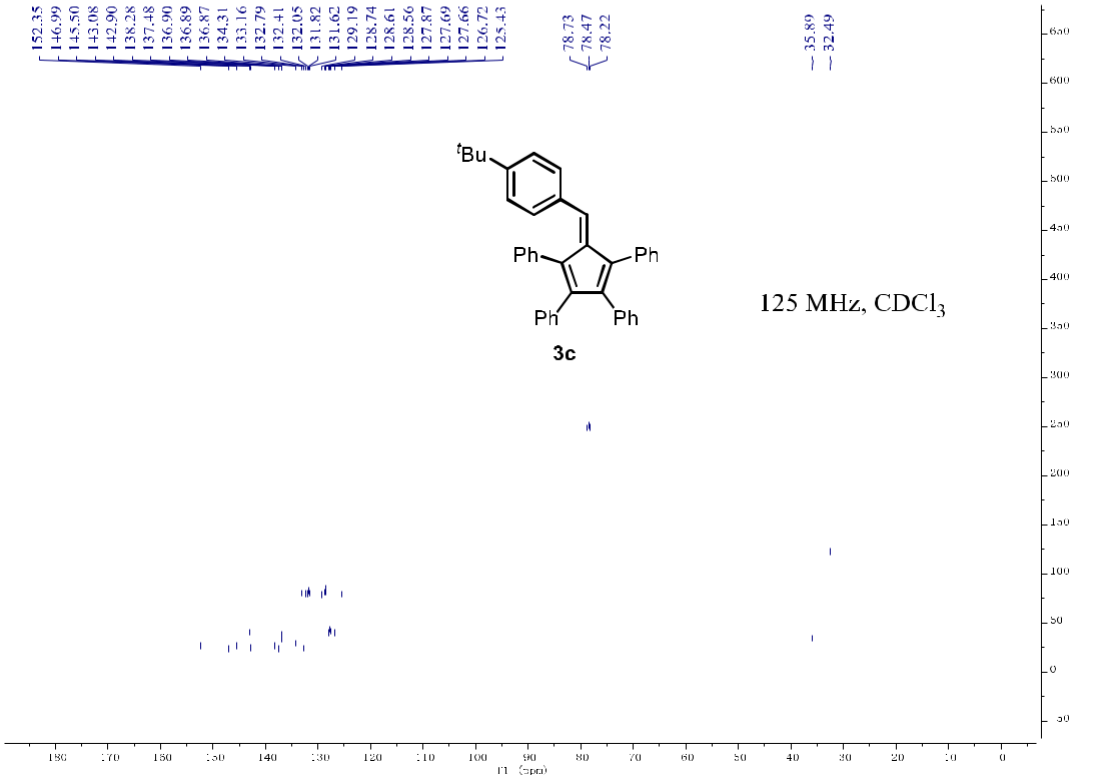


(5-(4-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3b)

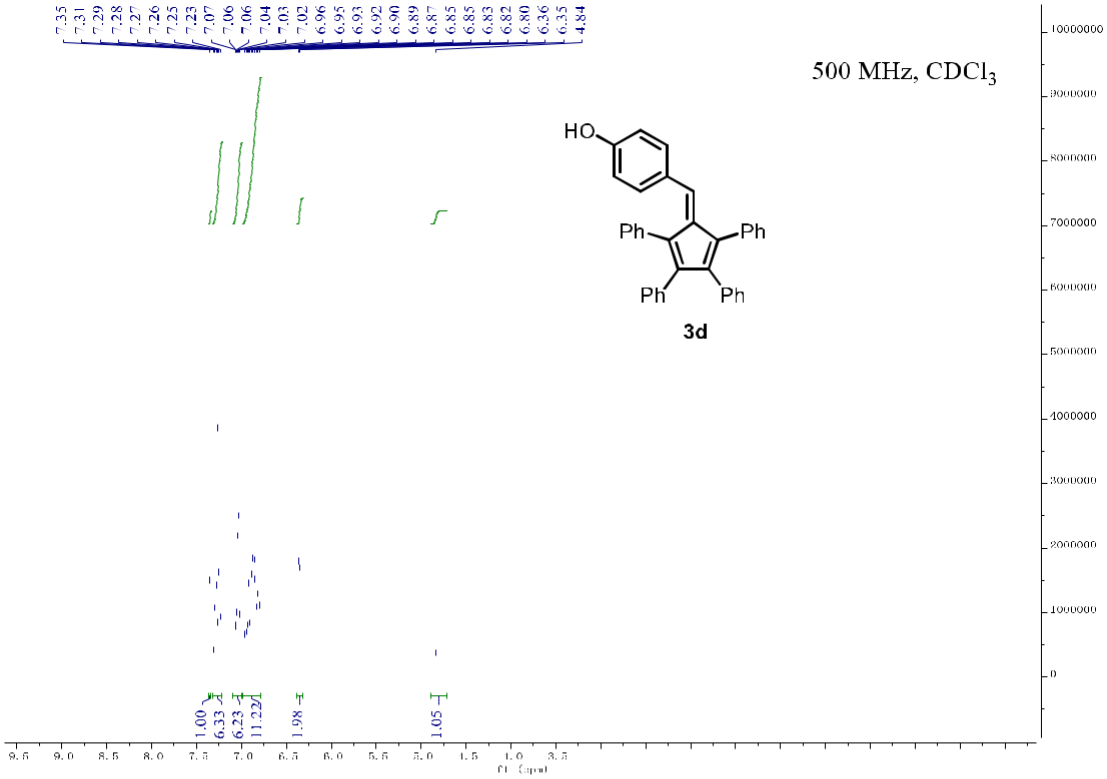


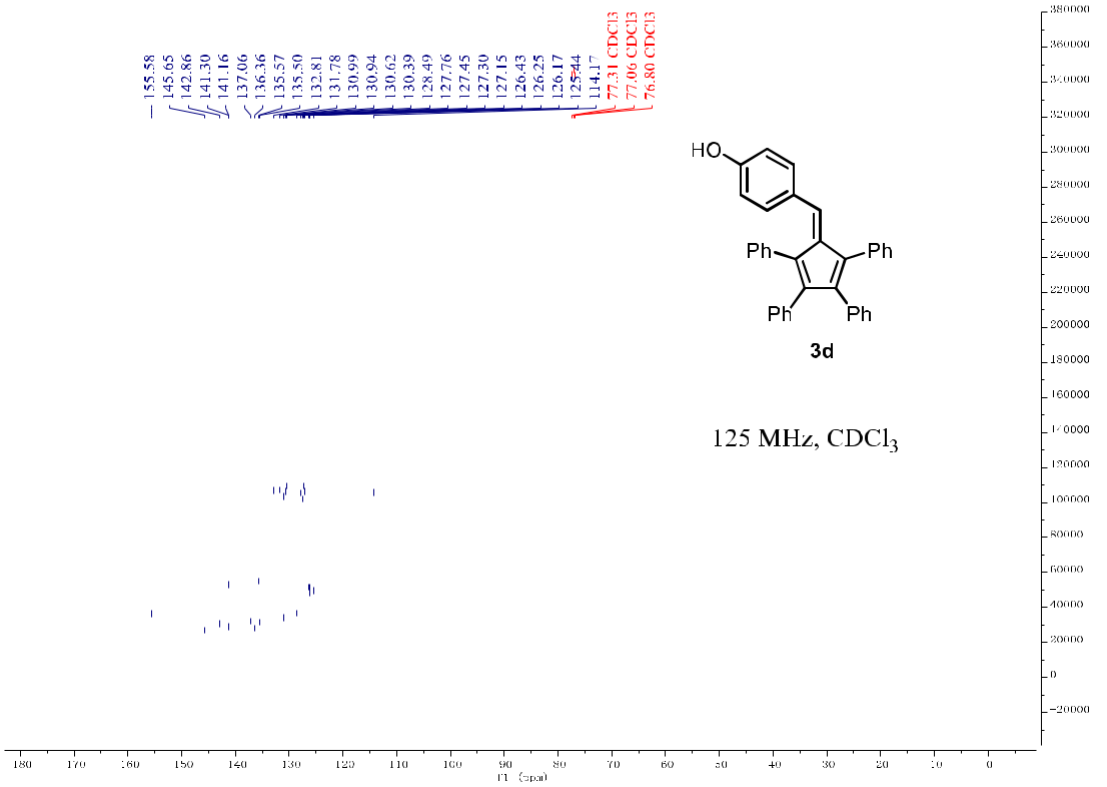
(5-(4-(tert-butyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene(3c)



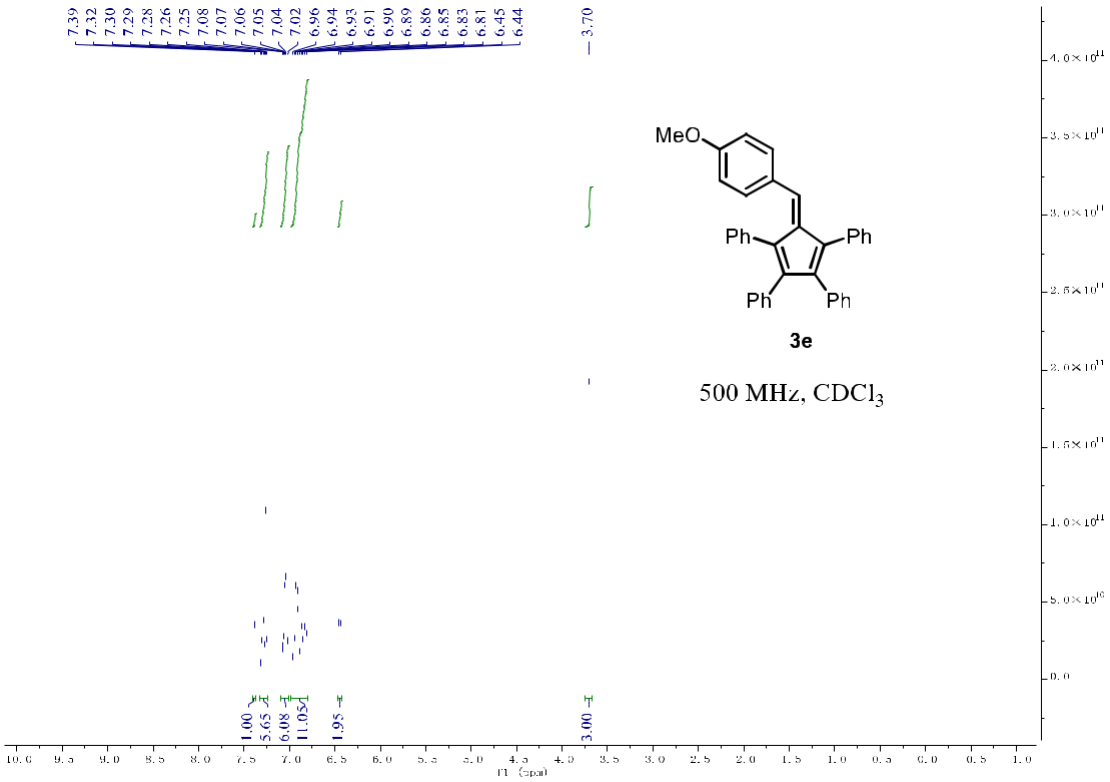


4-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenol (3d)

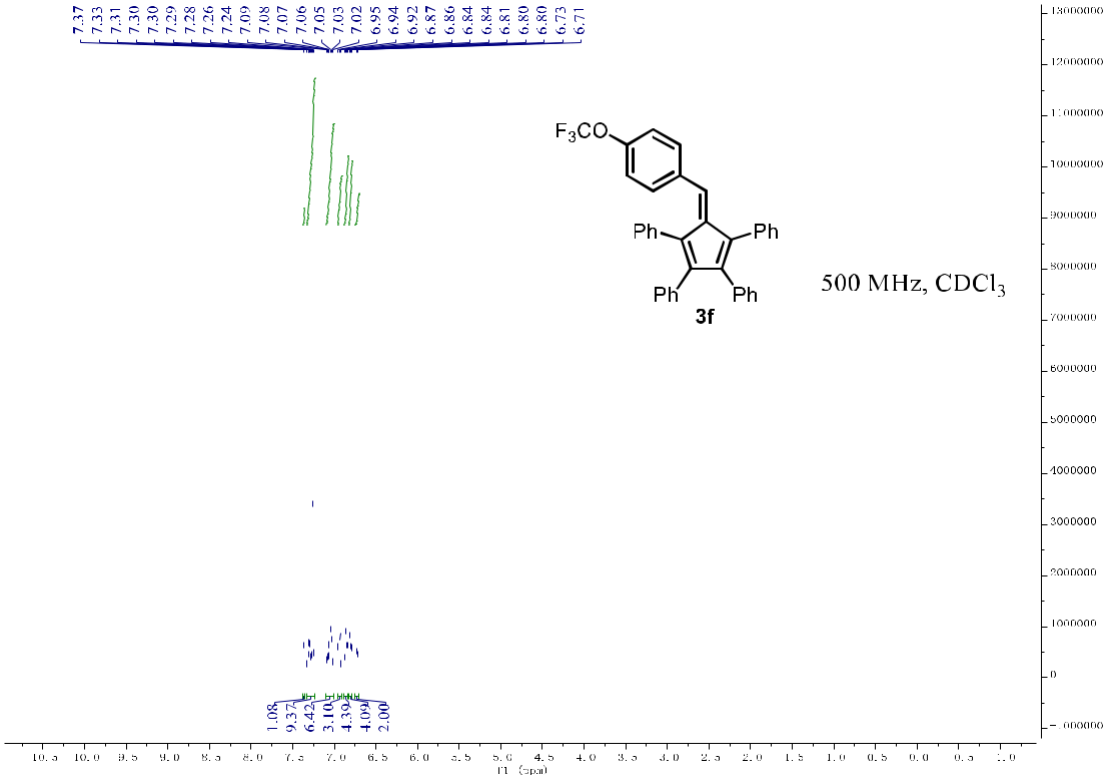


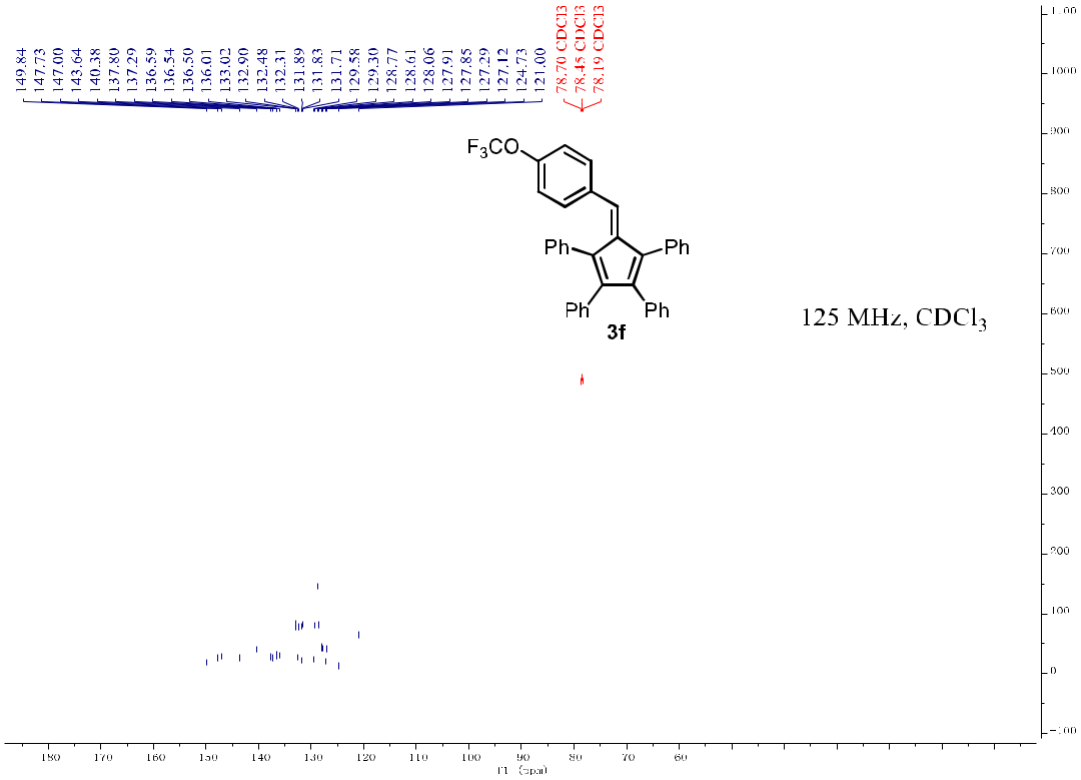


(5-(4-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3e)

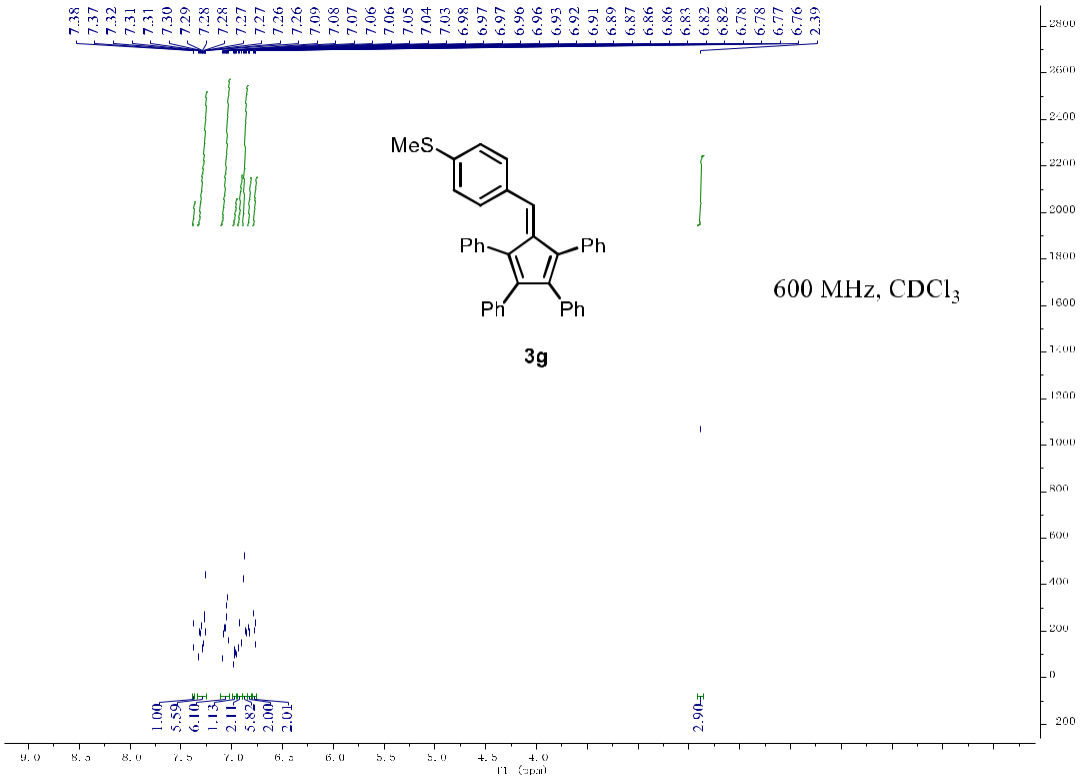


5-(4-(trifluoromethoxy)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3f)

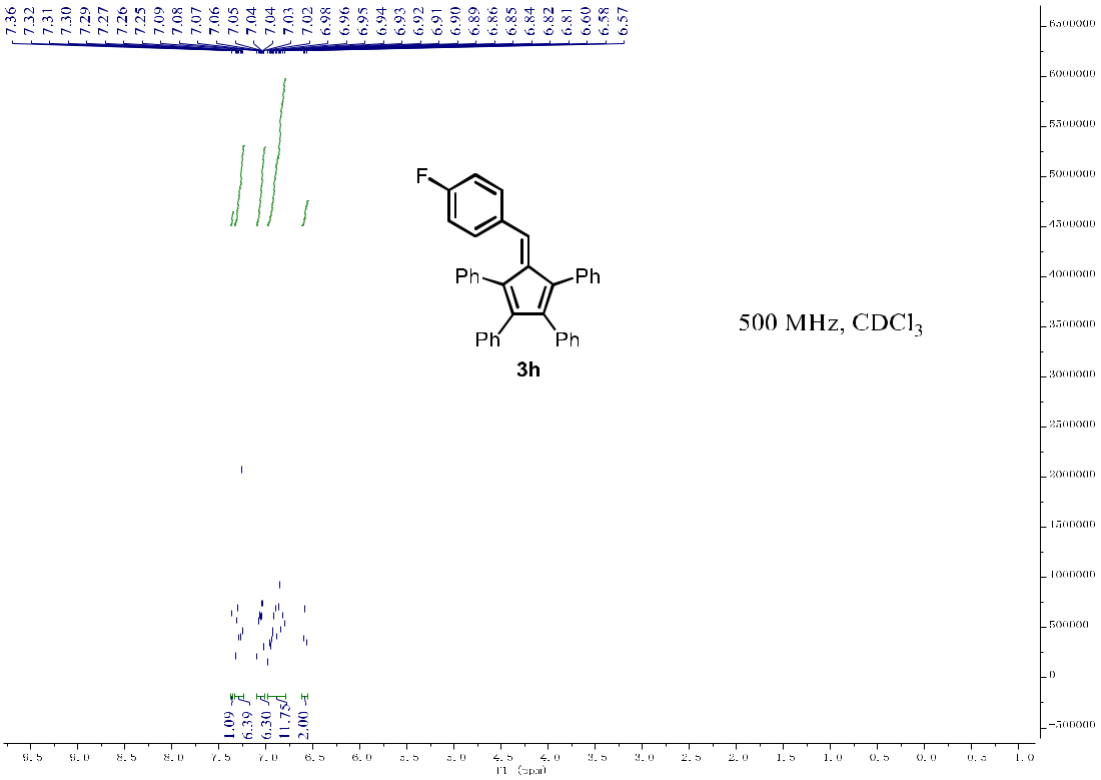




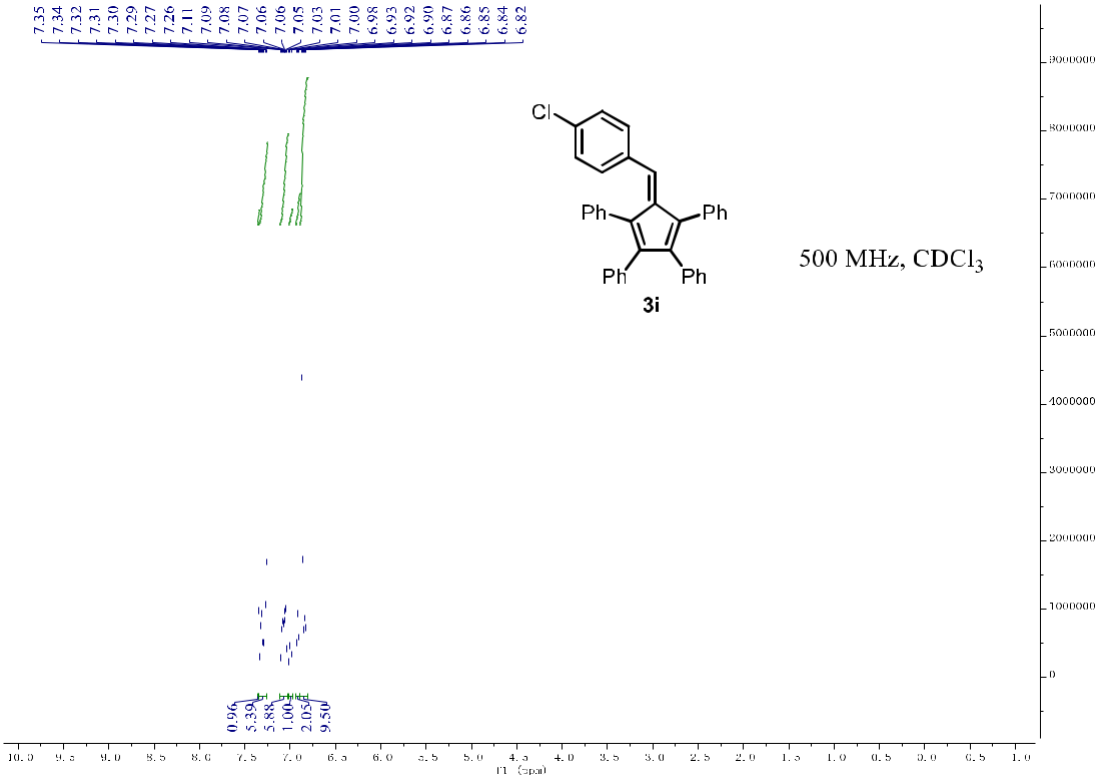
methyl(4-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)sulfane (3g)

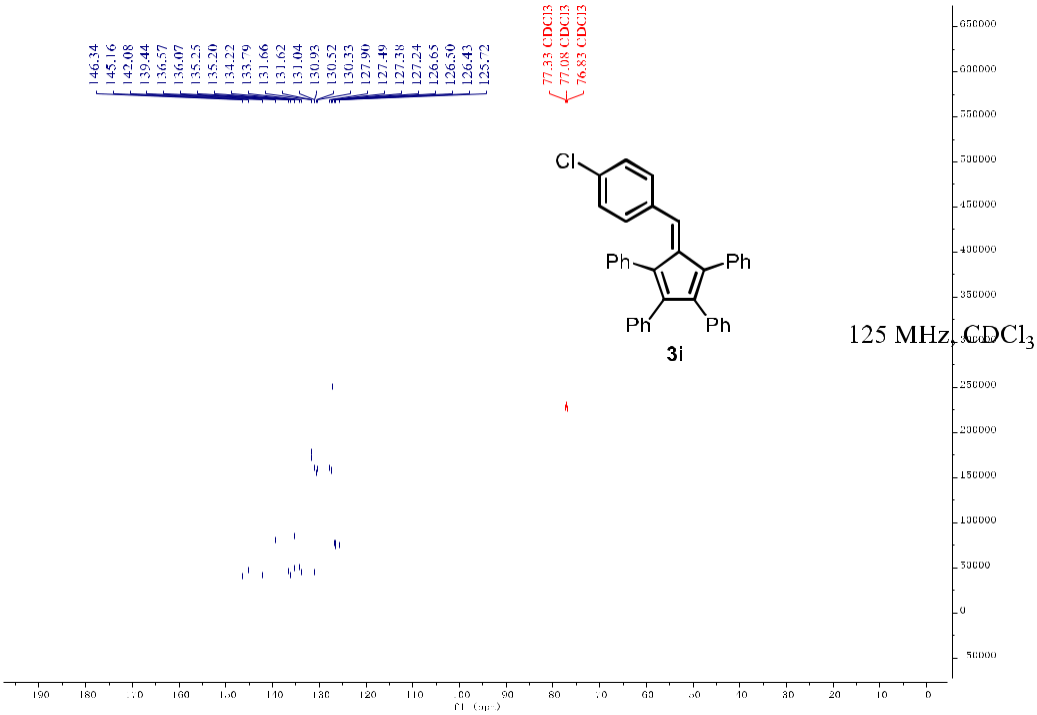


(5-(4-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3h)

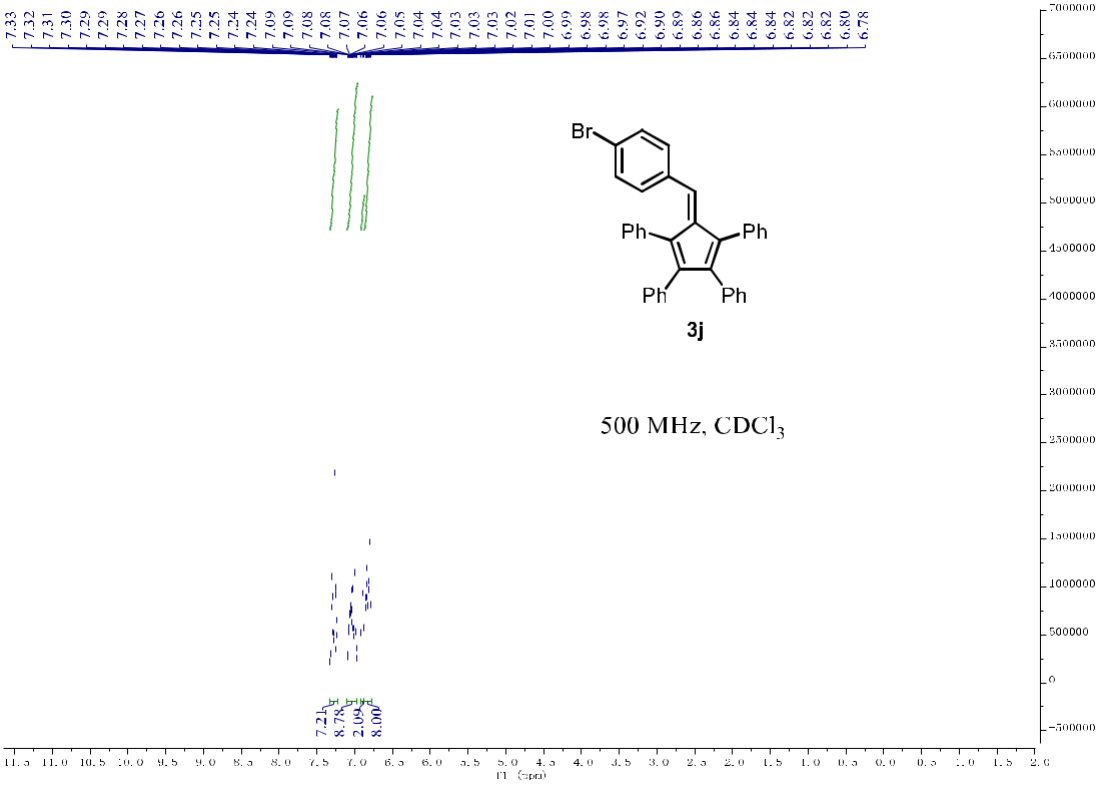


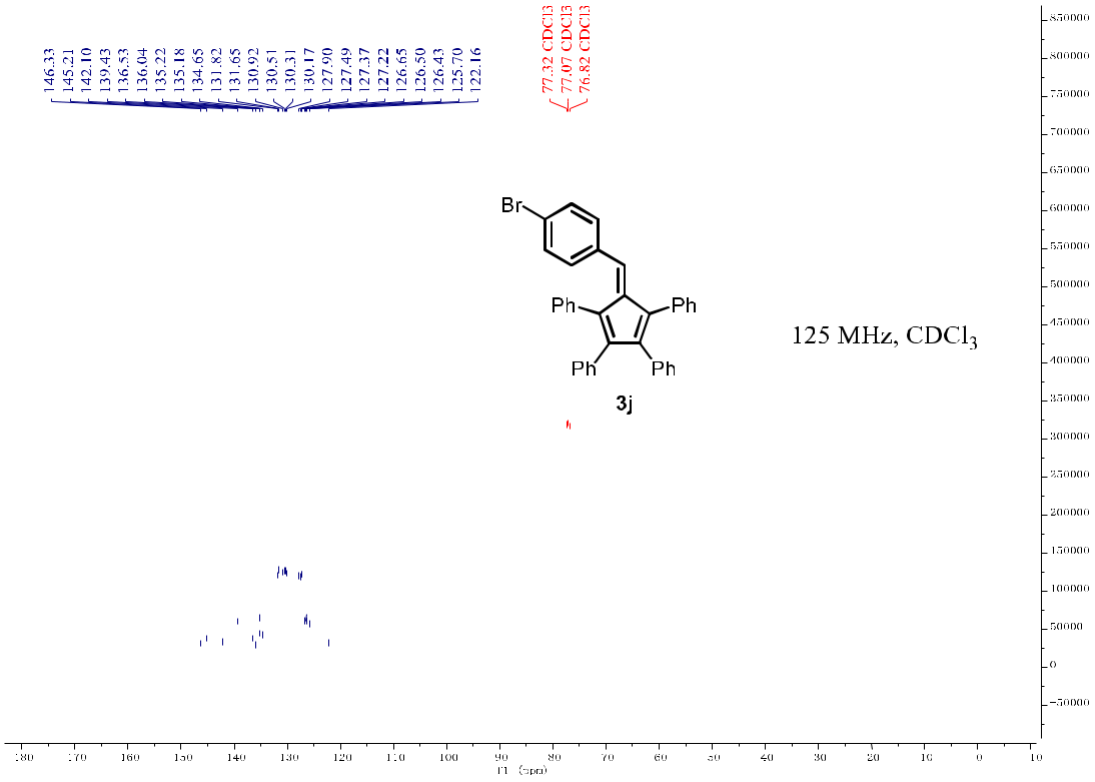
(5-(4-chlorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3i)



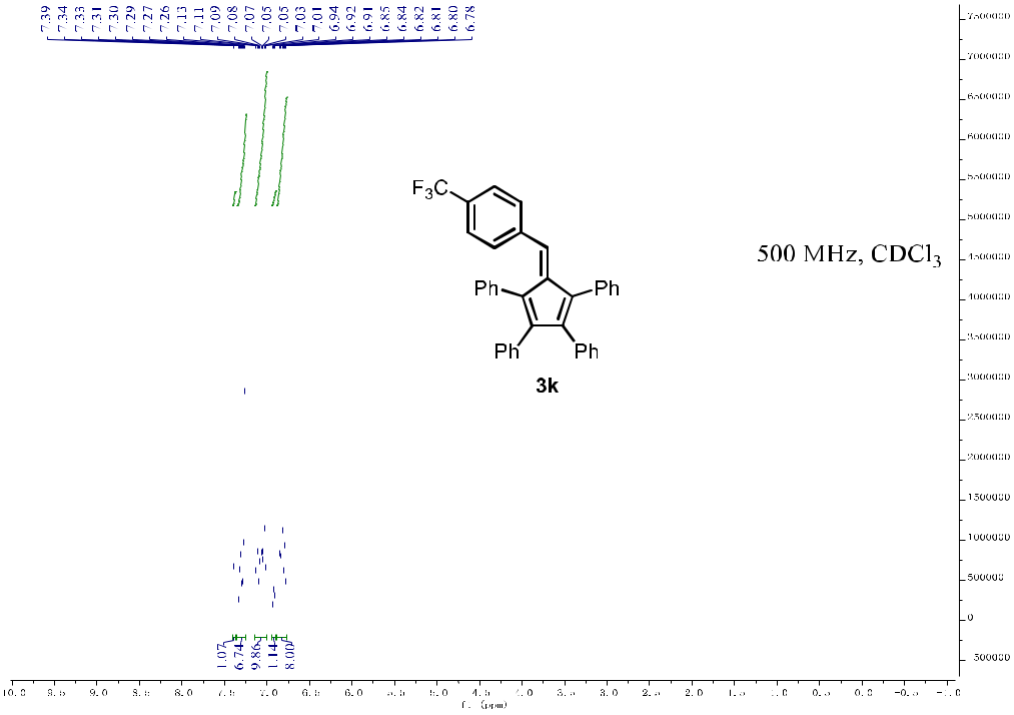


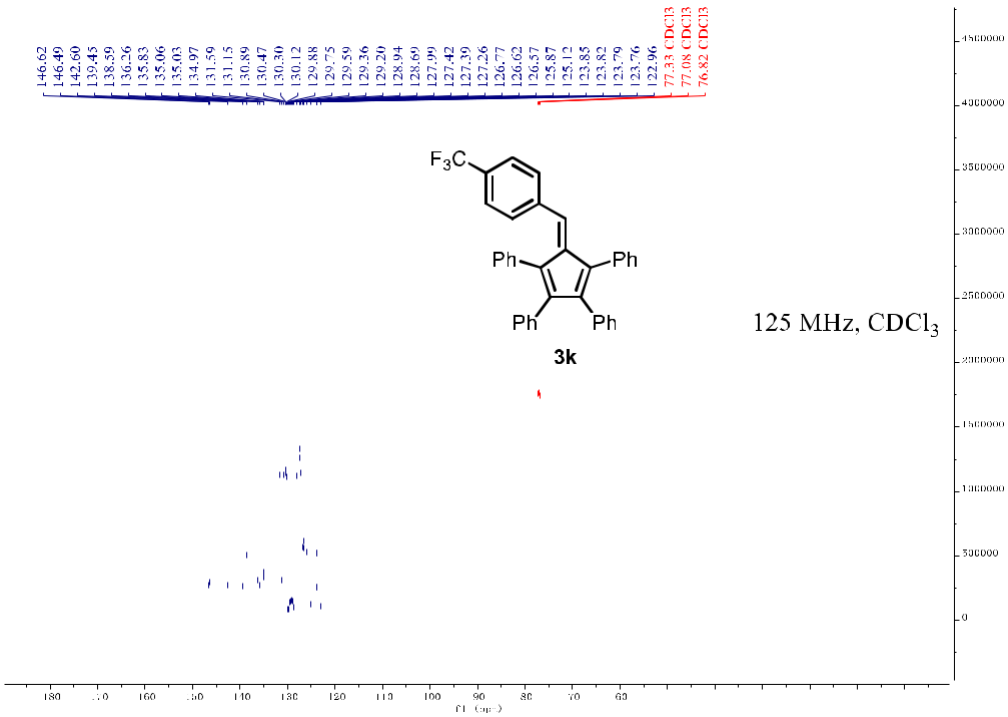
(5-(4-bromobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3j)



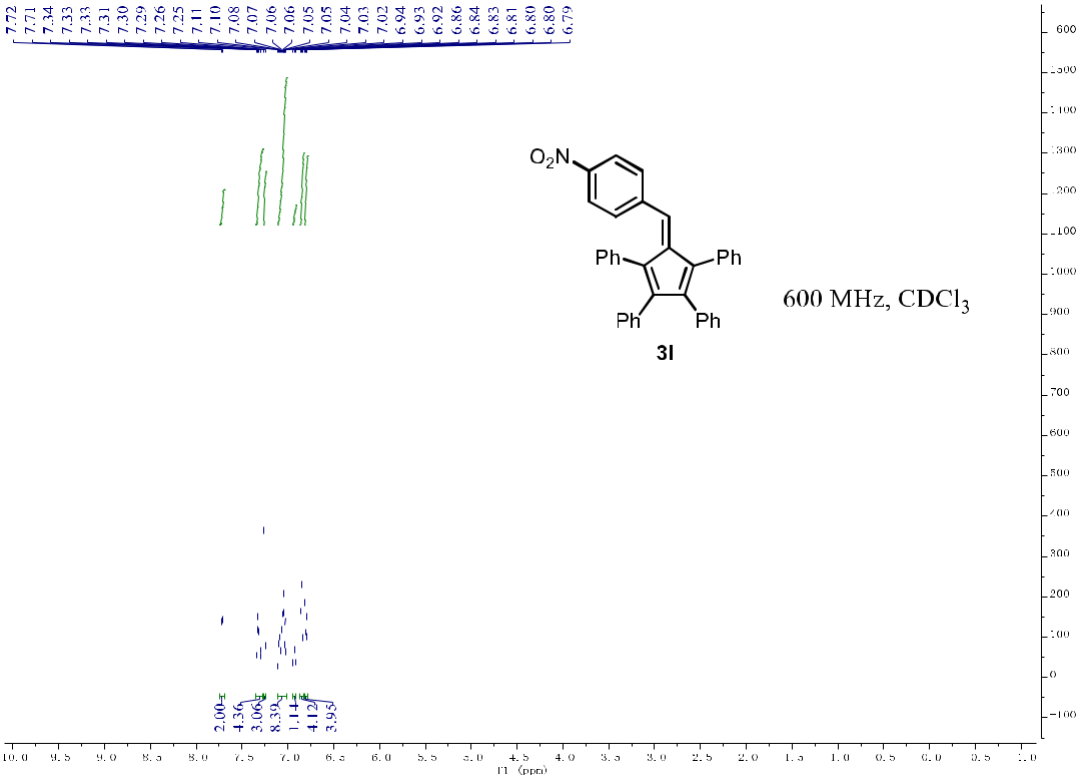


(5-(4-(trifluoromethyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3k)

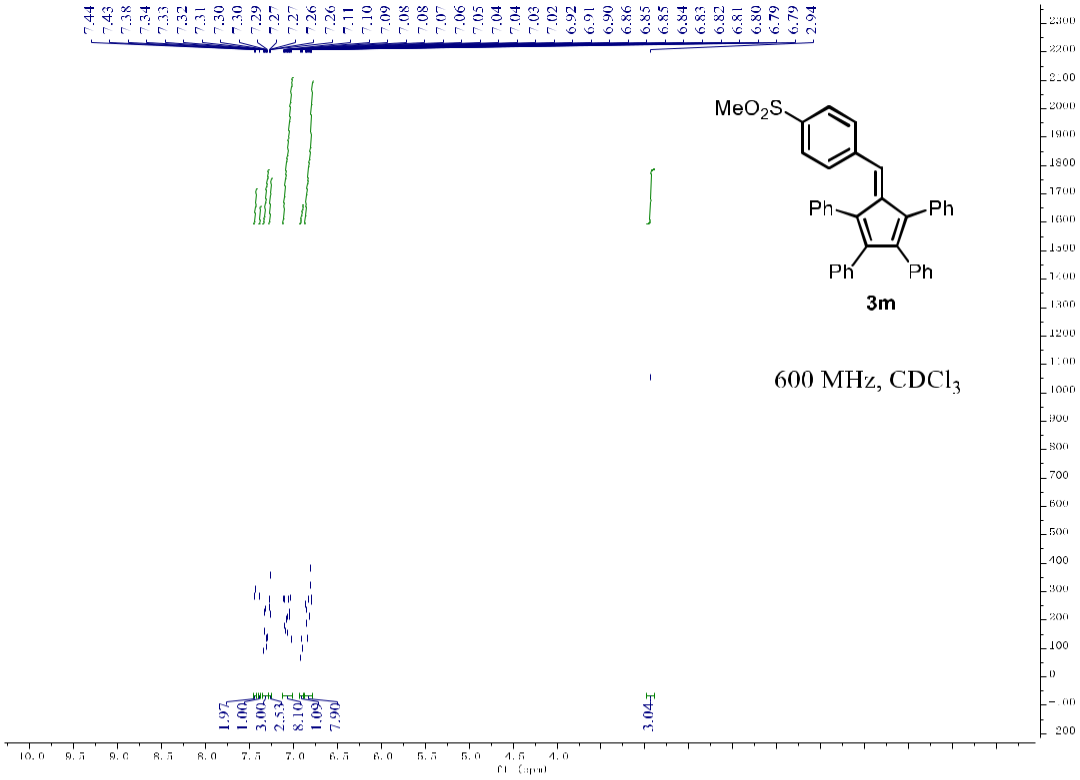


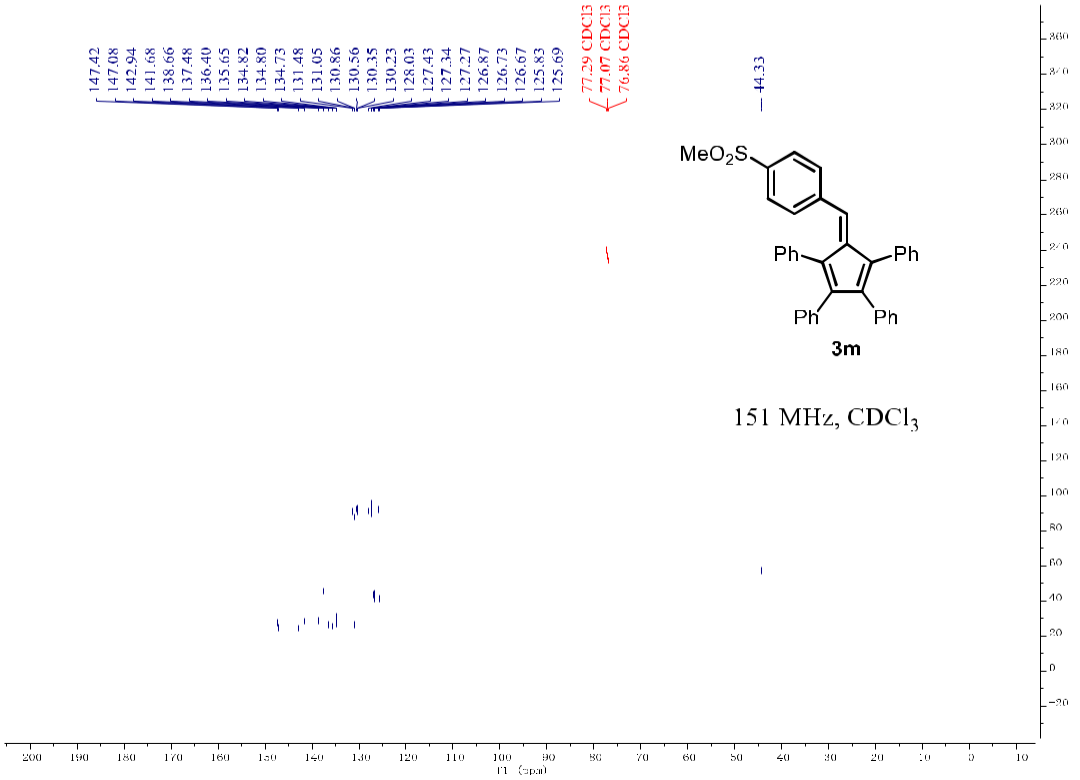


(5-(4-nitrobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3l)

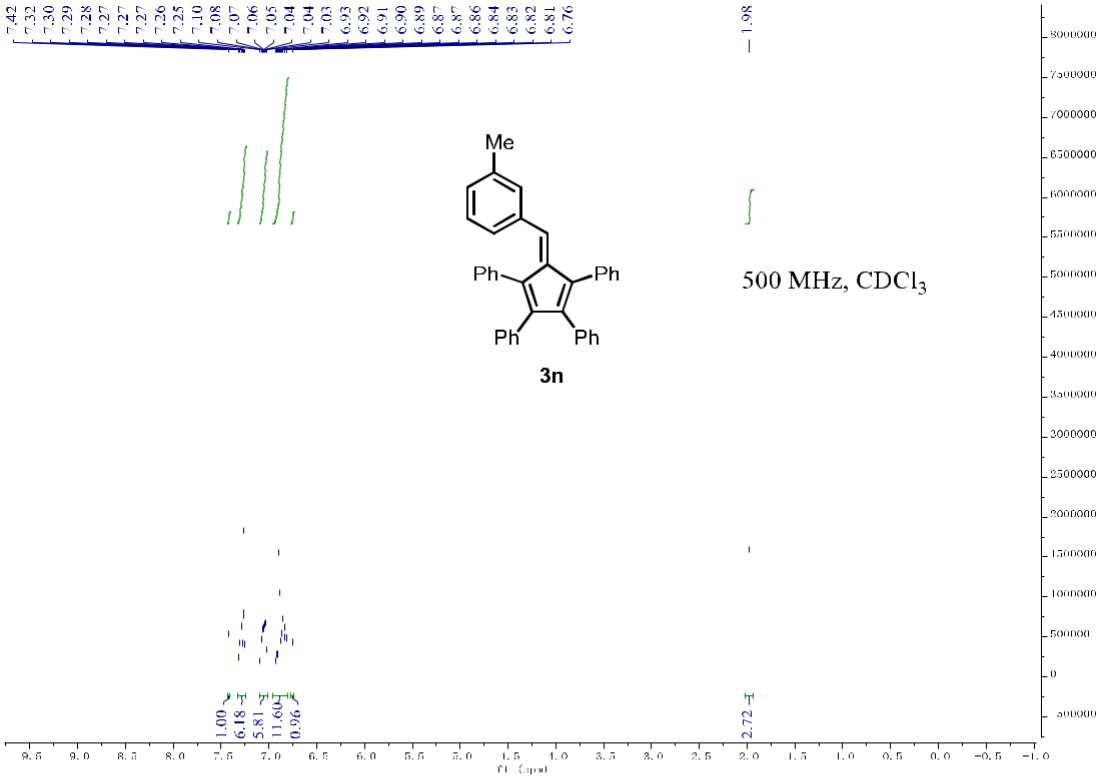


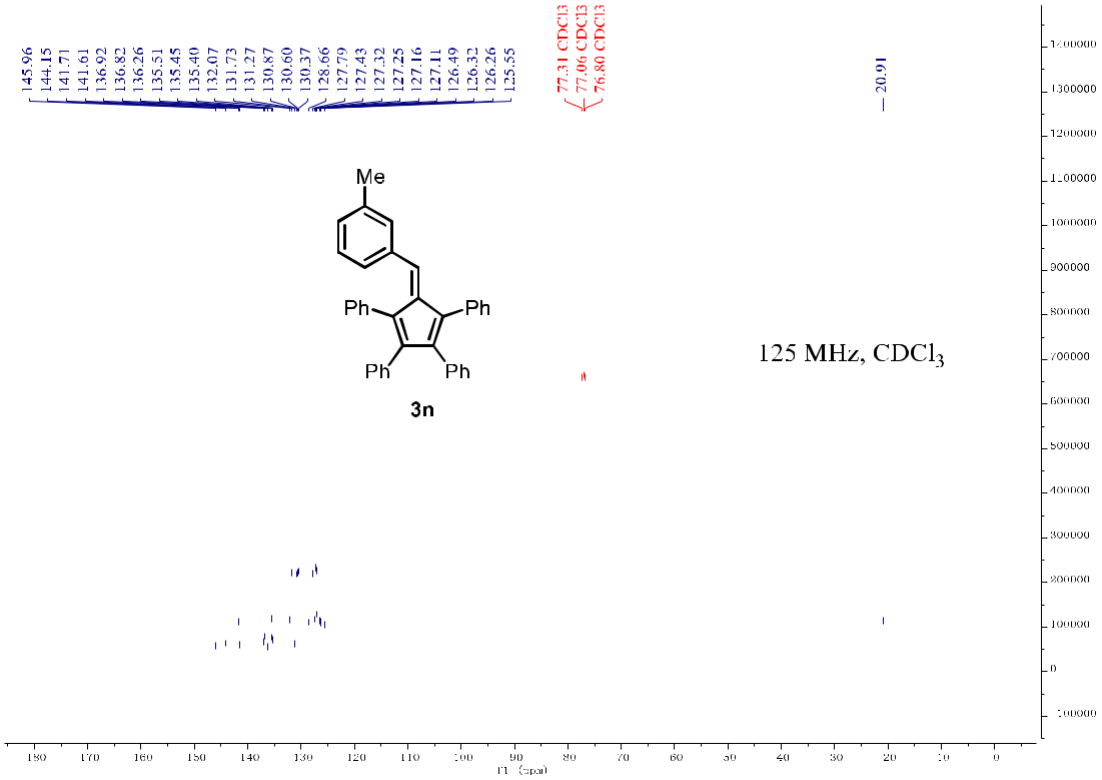
(5-(4-(methylsulfonyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3m)



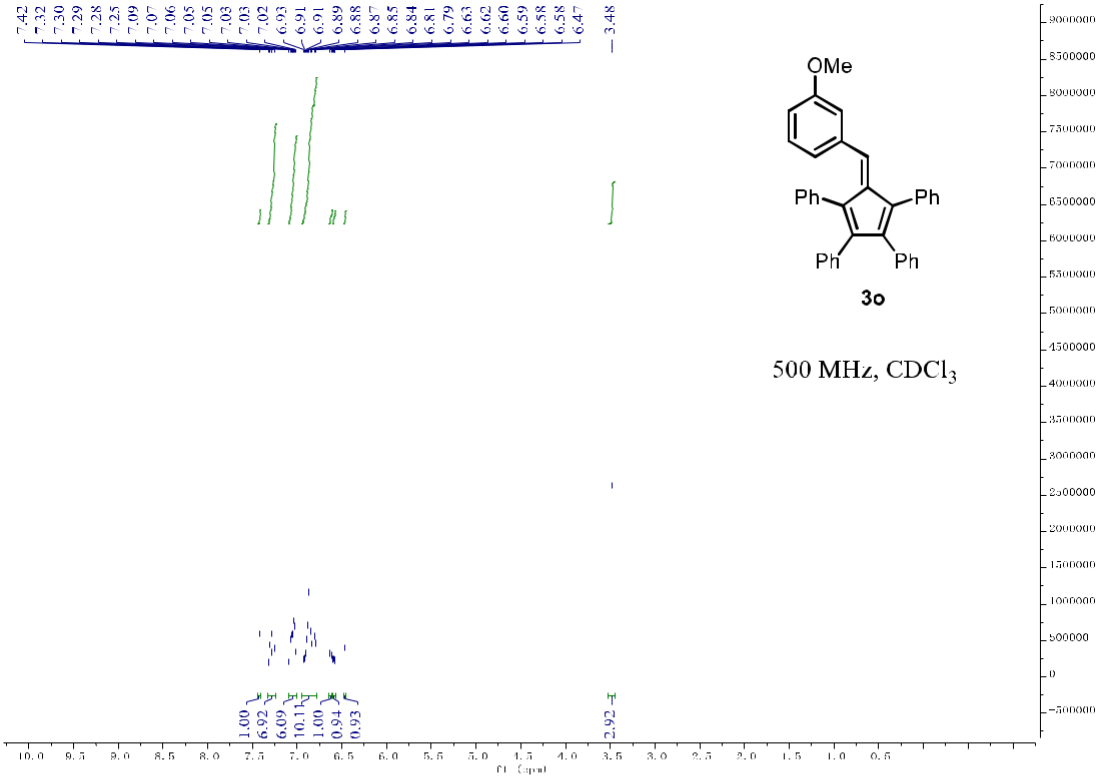


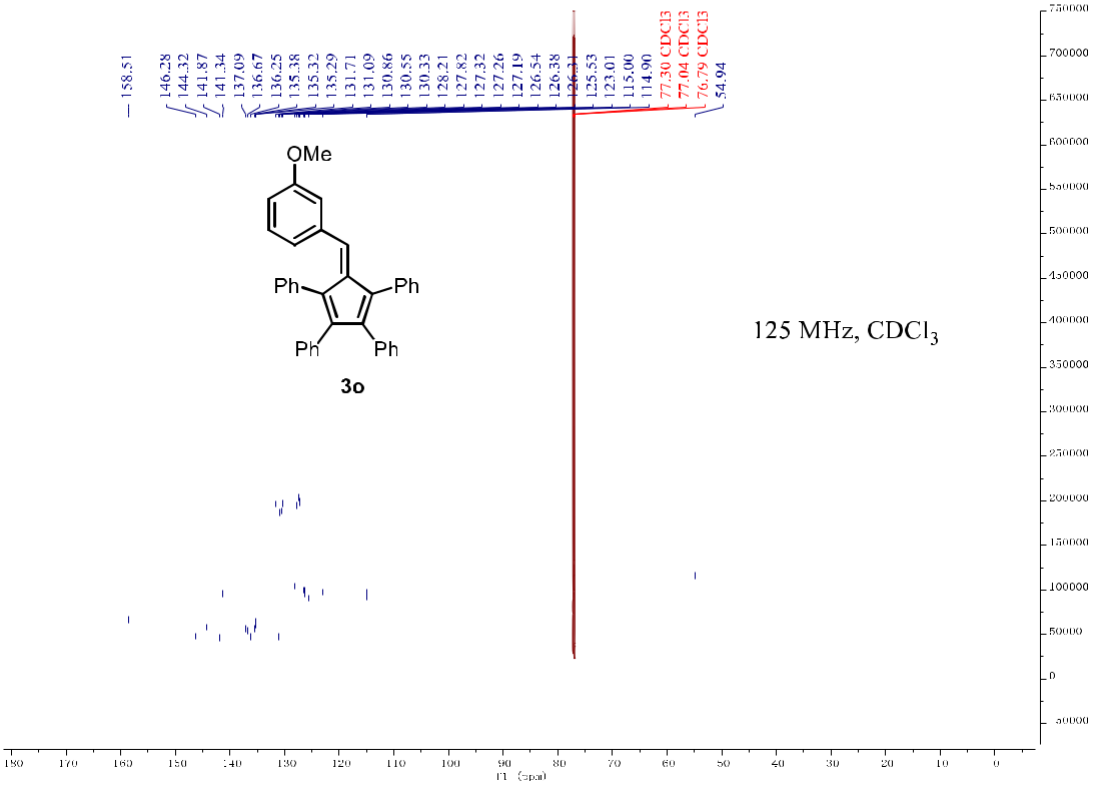
(5-(3-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3n)



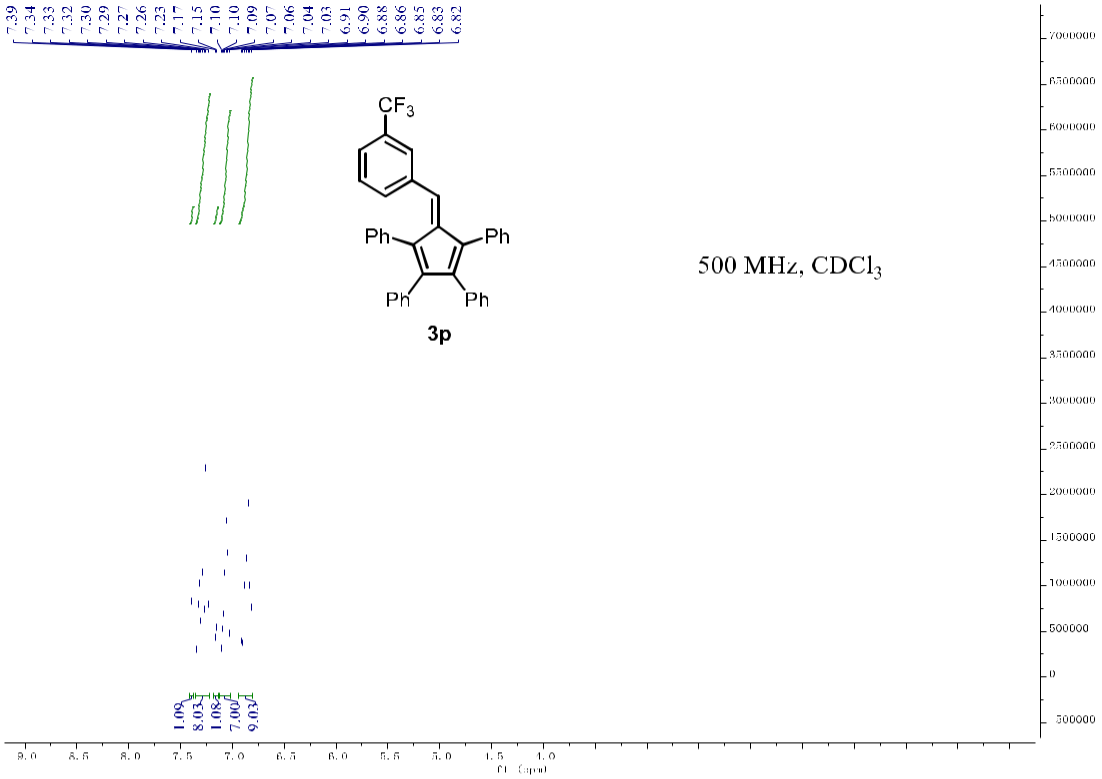


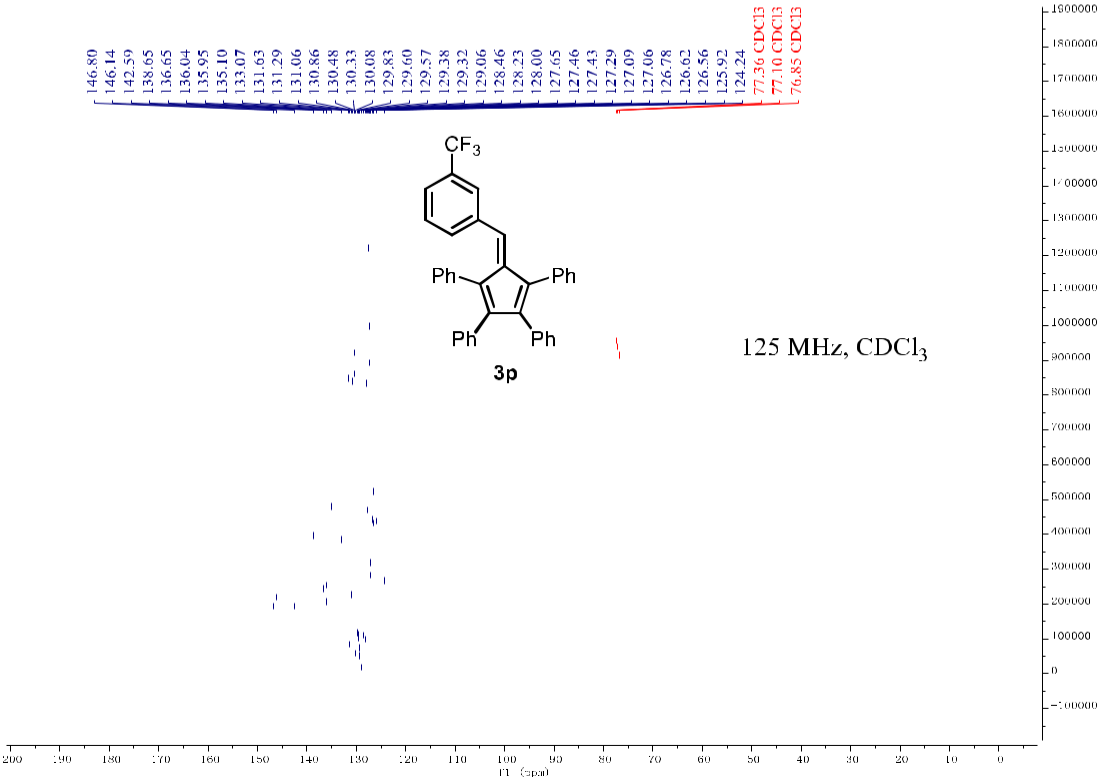
(5-(3-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3o)



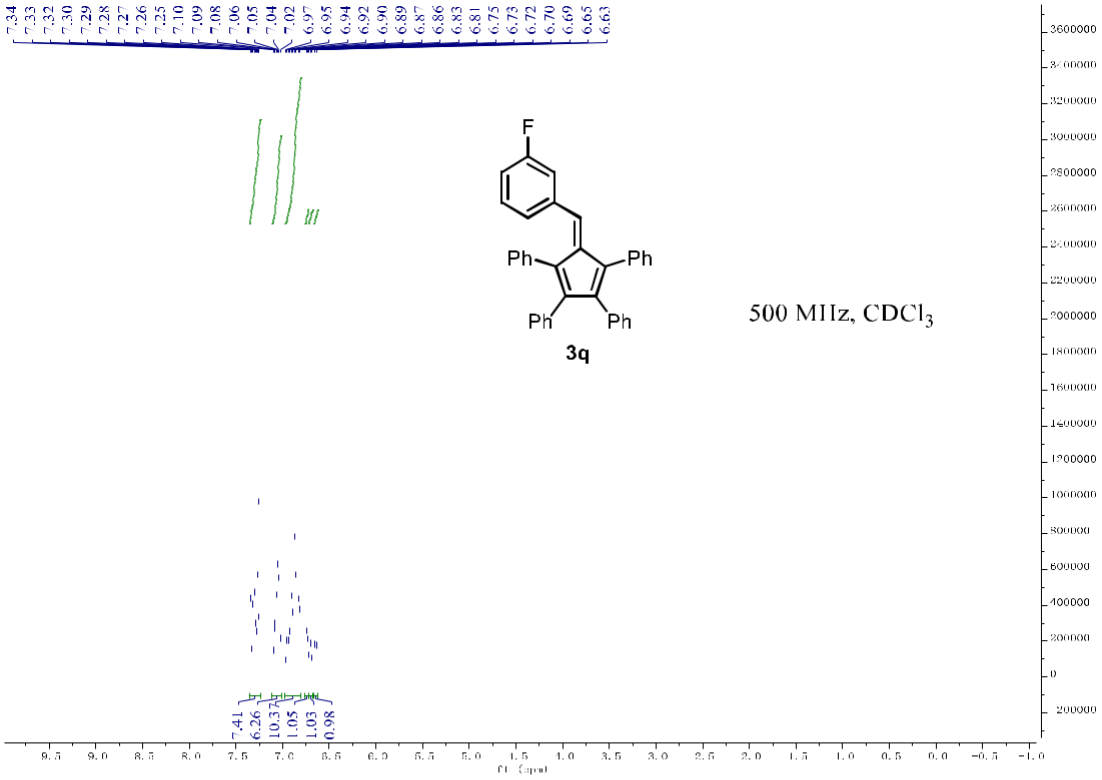


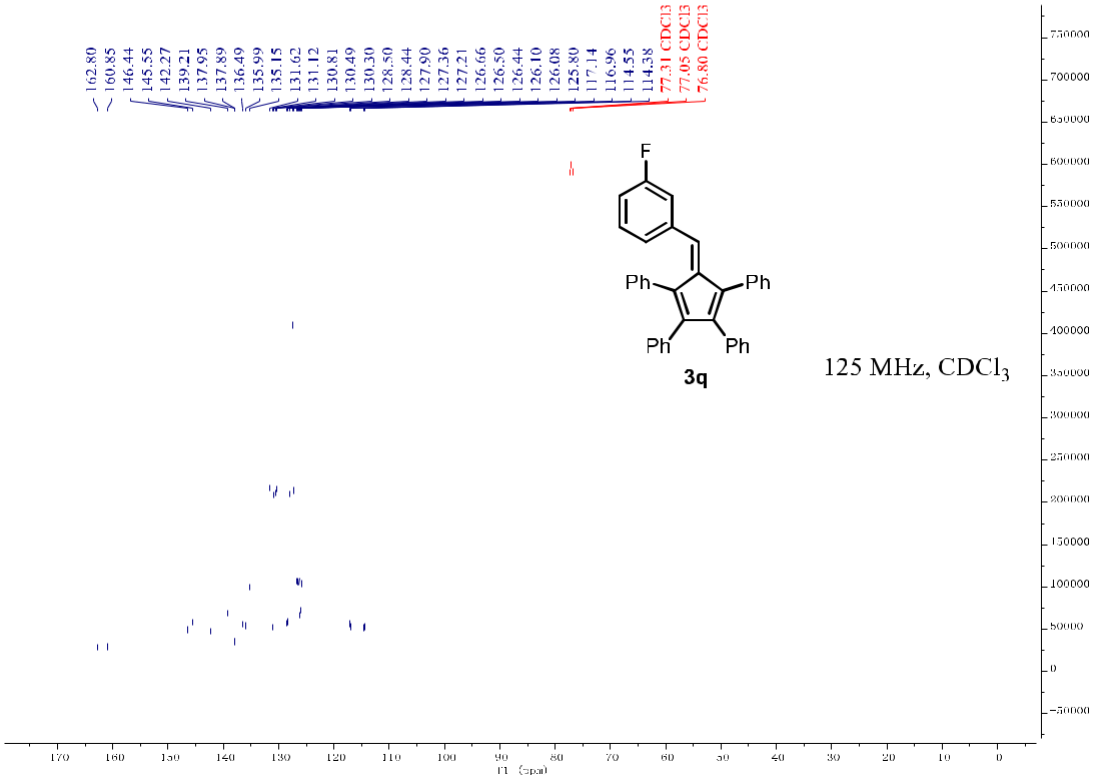
(5-(3-(trifluoromethyl)benzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3p)



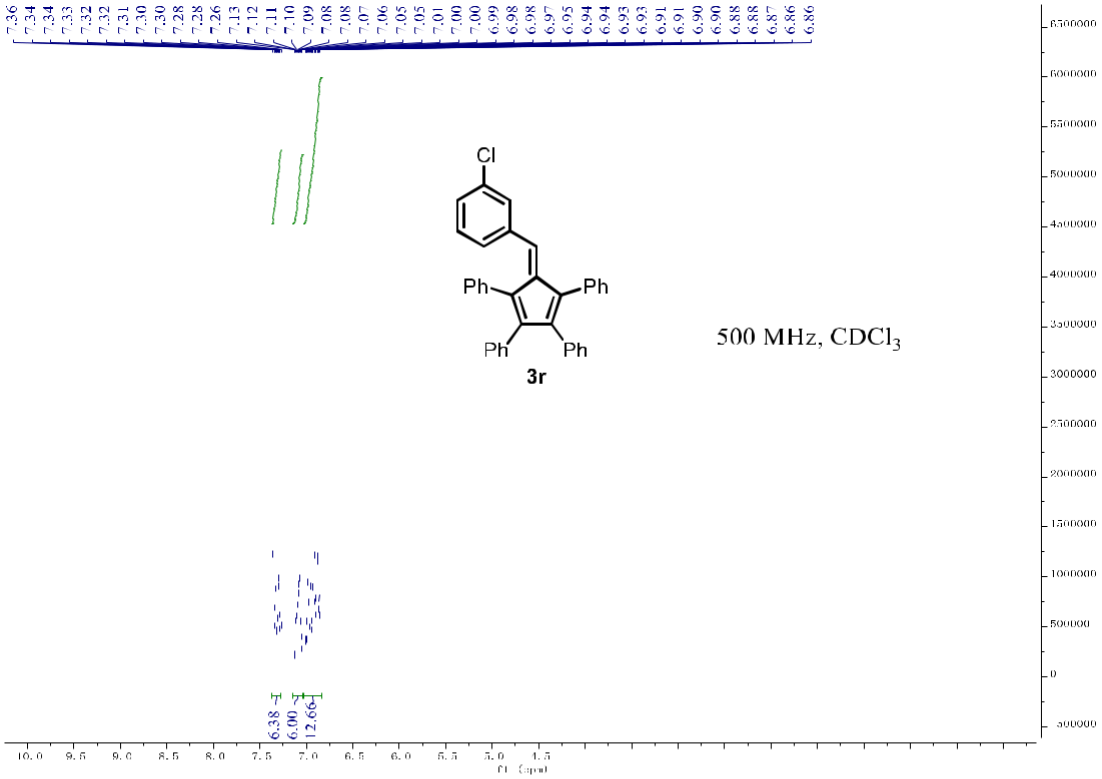


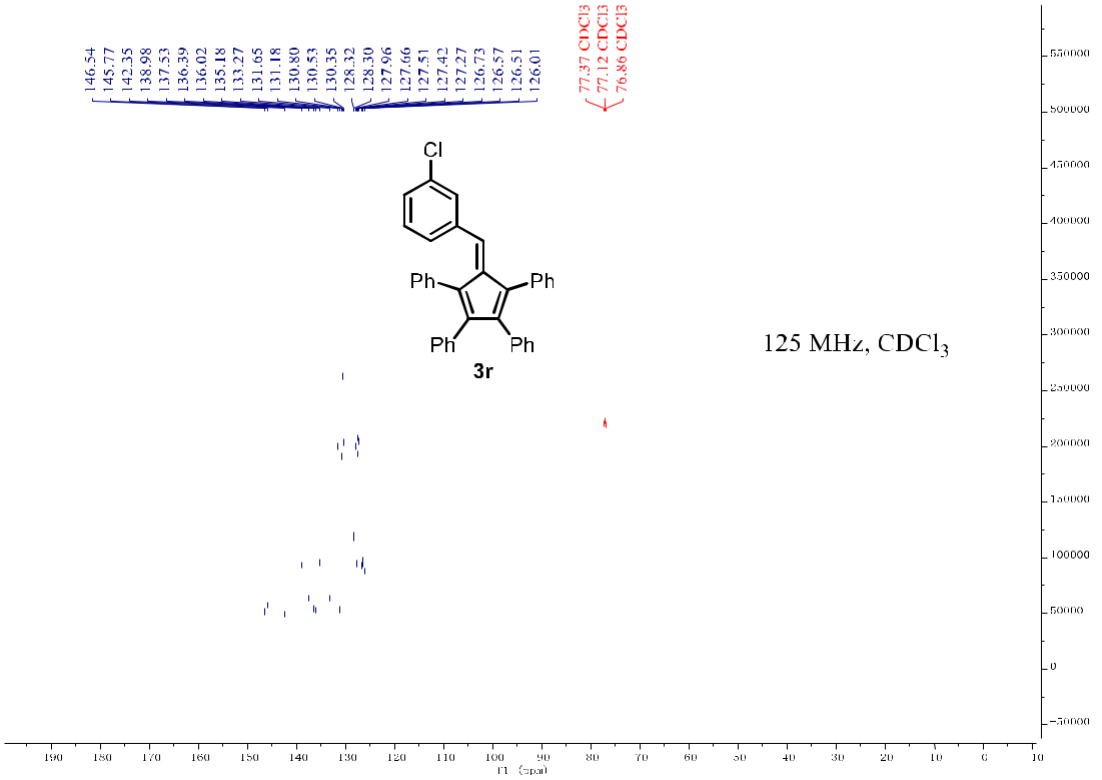
(5-(3-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3q)



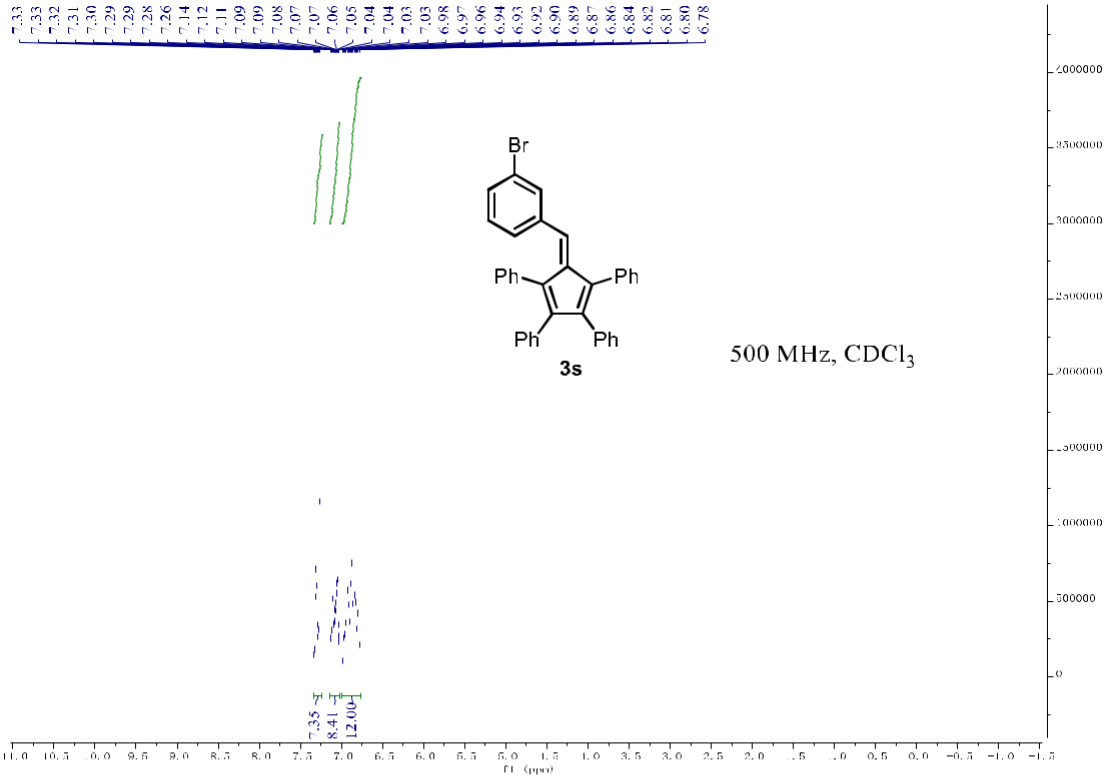


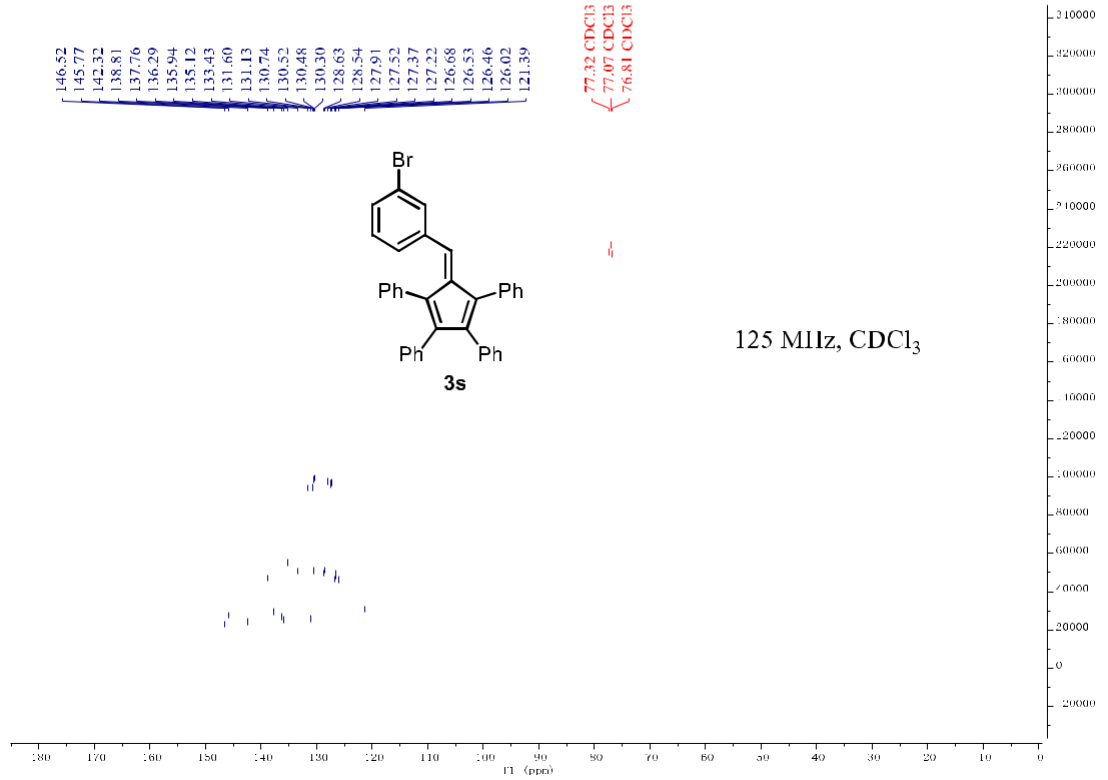
(5-(3-chlorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3r)



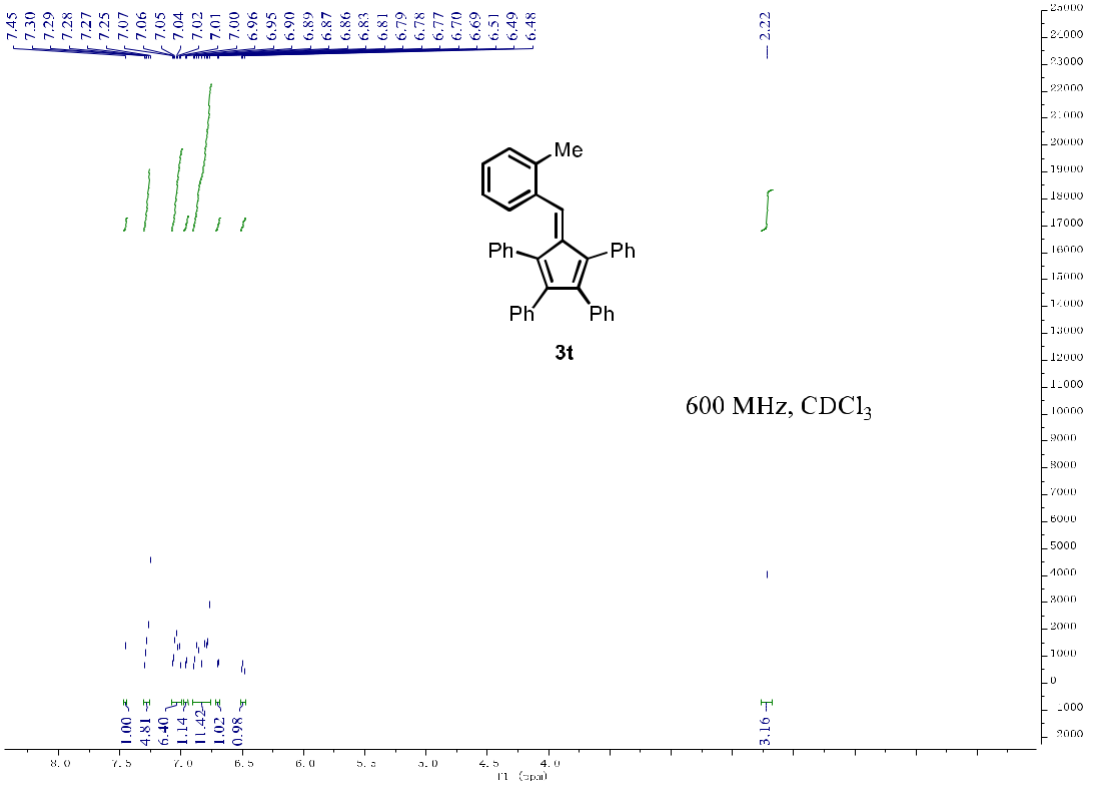


(5-(3-bromobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3s)

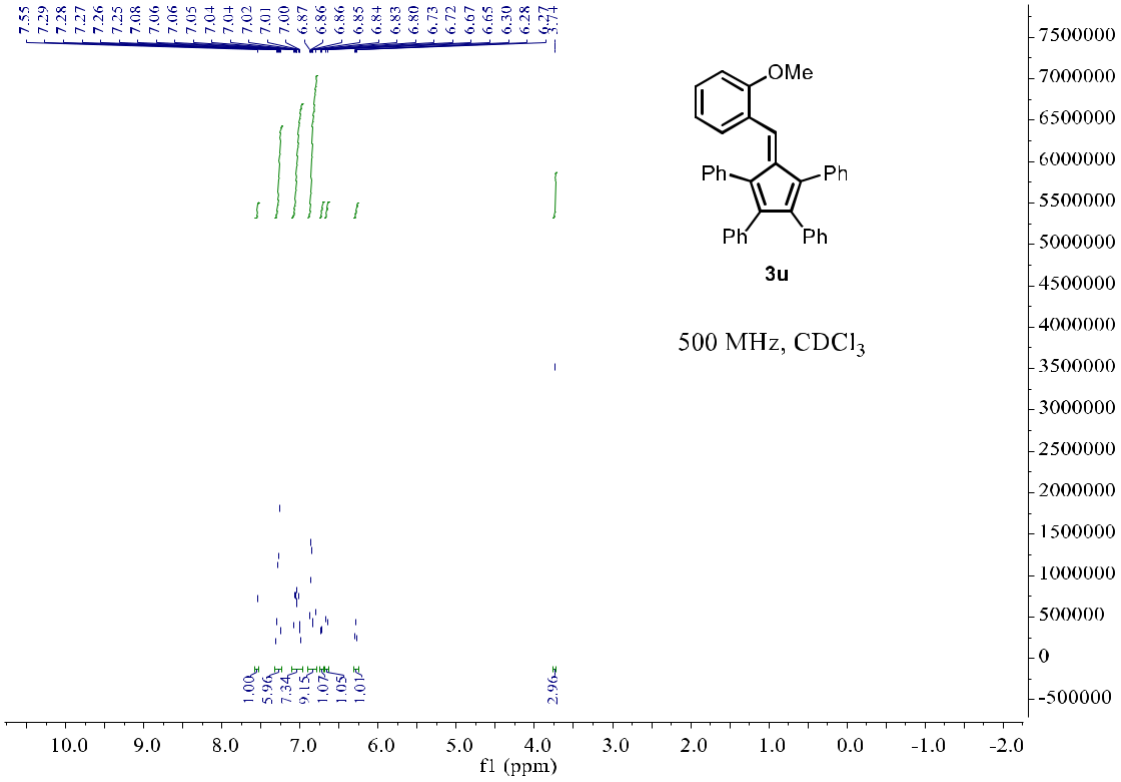




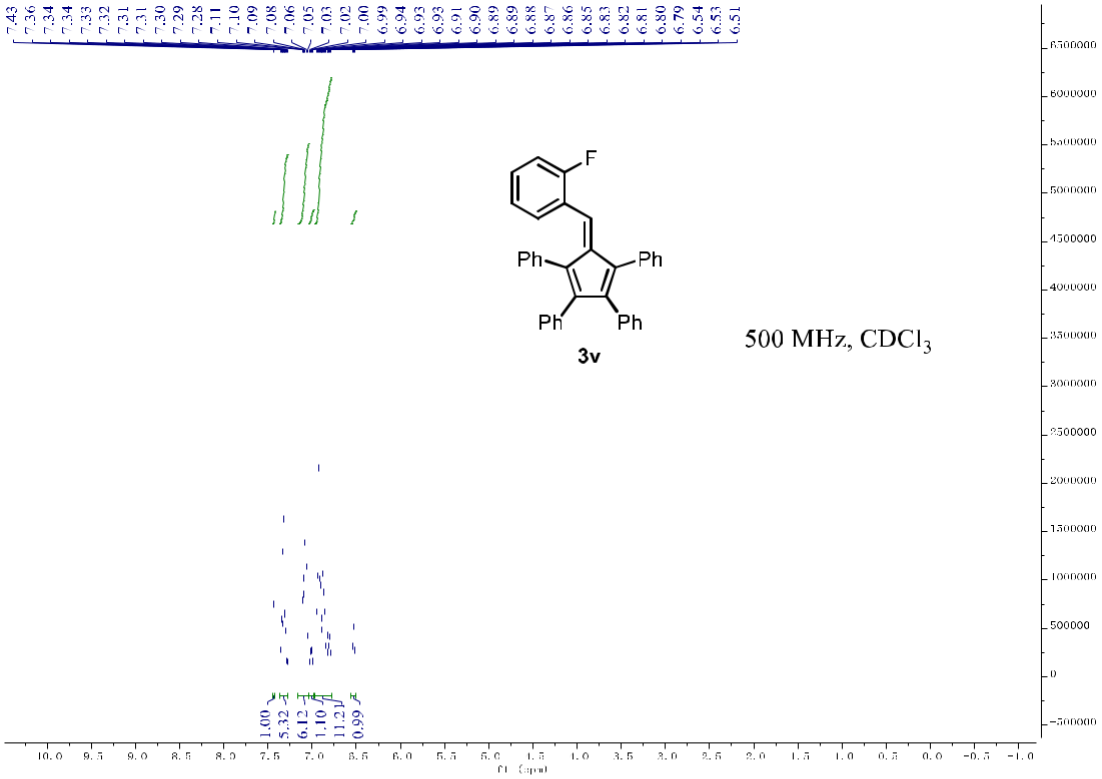
(5-(2-methylbenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3t)

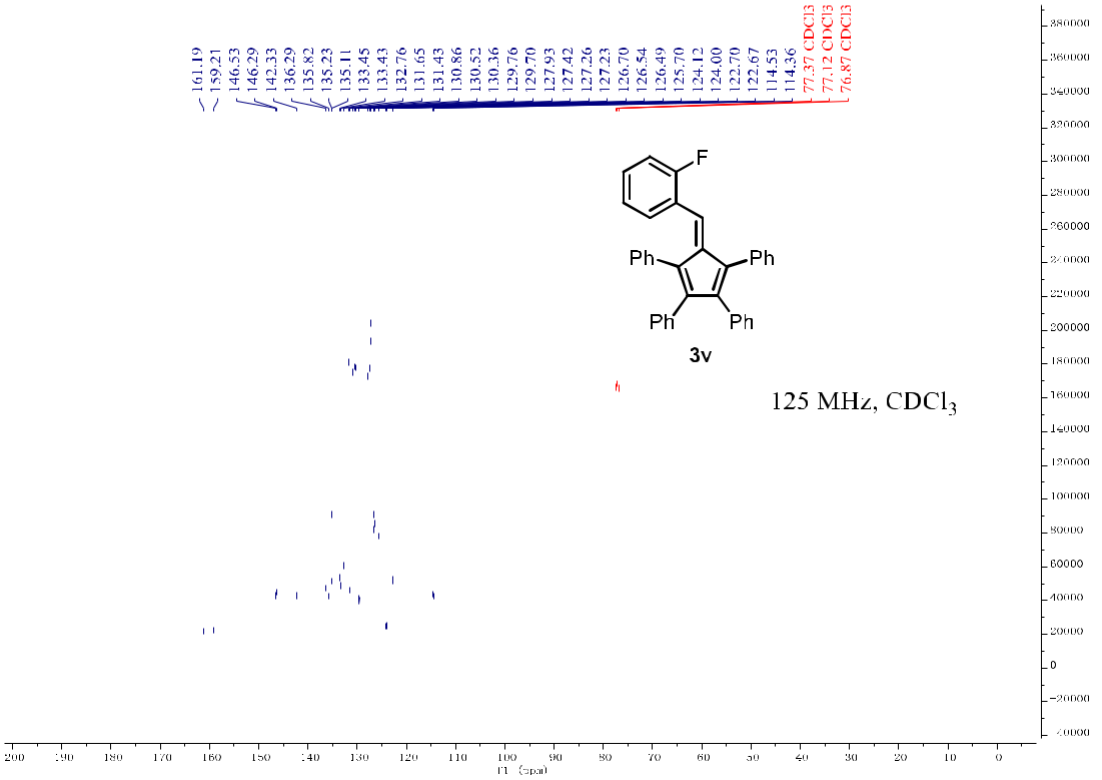


(5-(2-methoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3u)

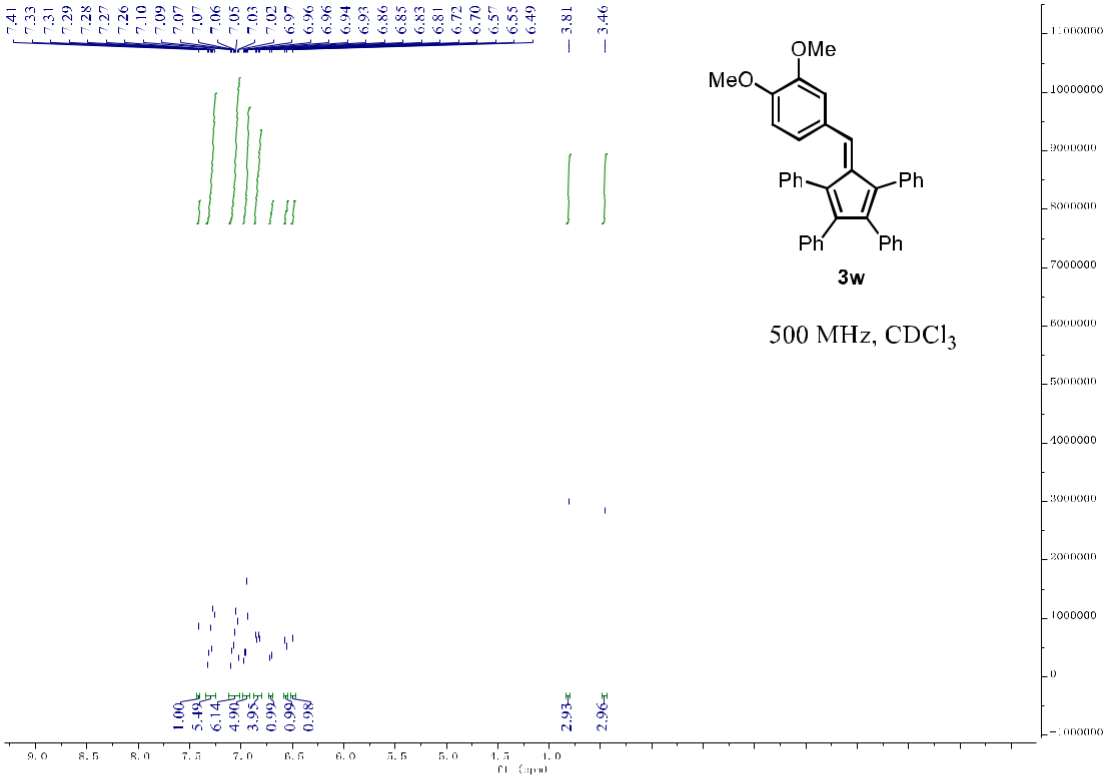


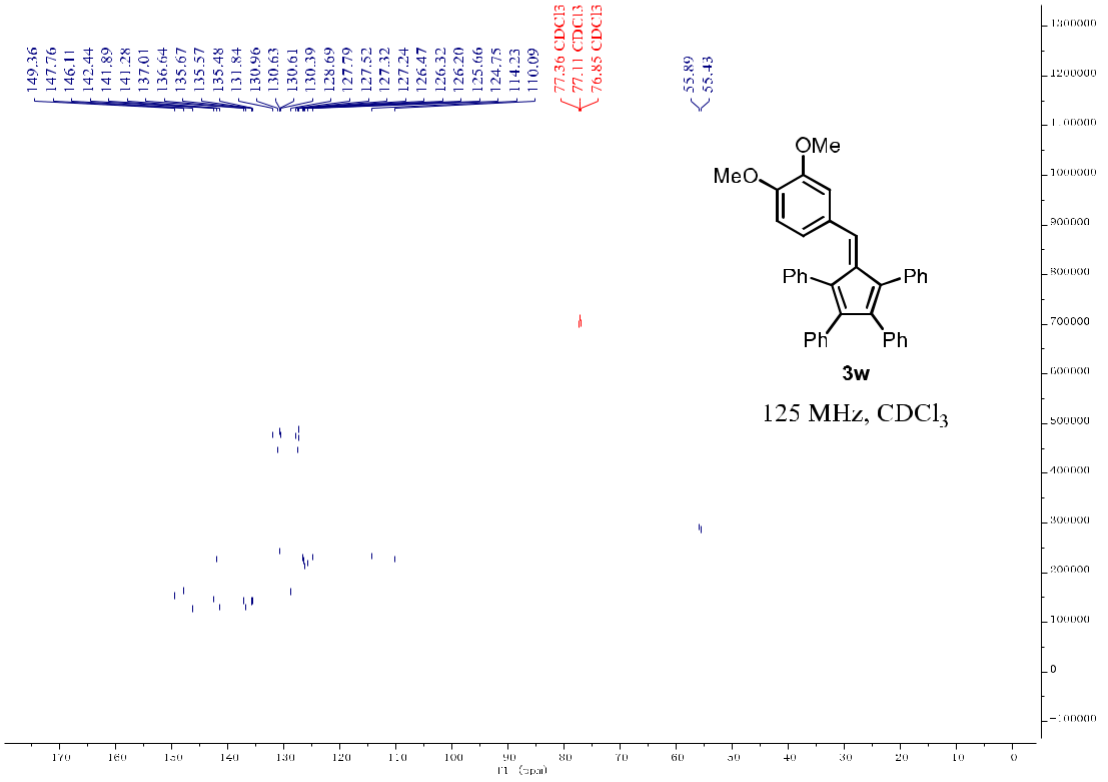
(5-(2-fluorobenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3v)



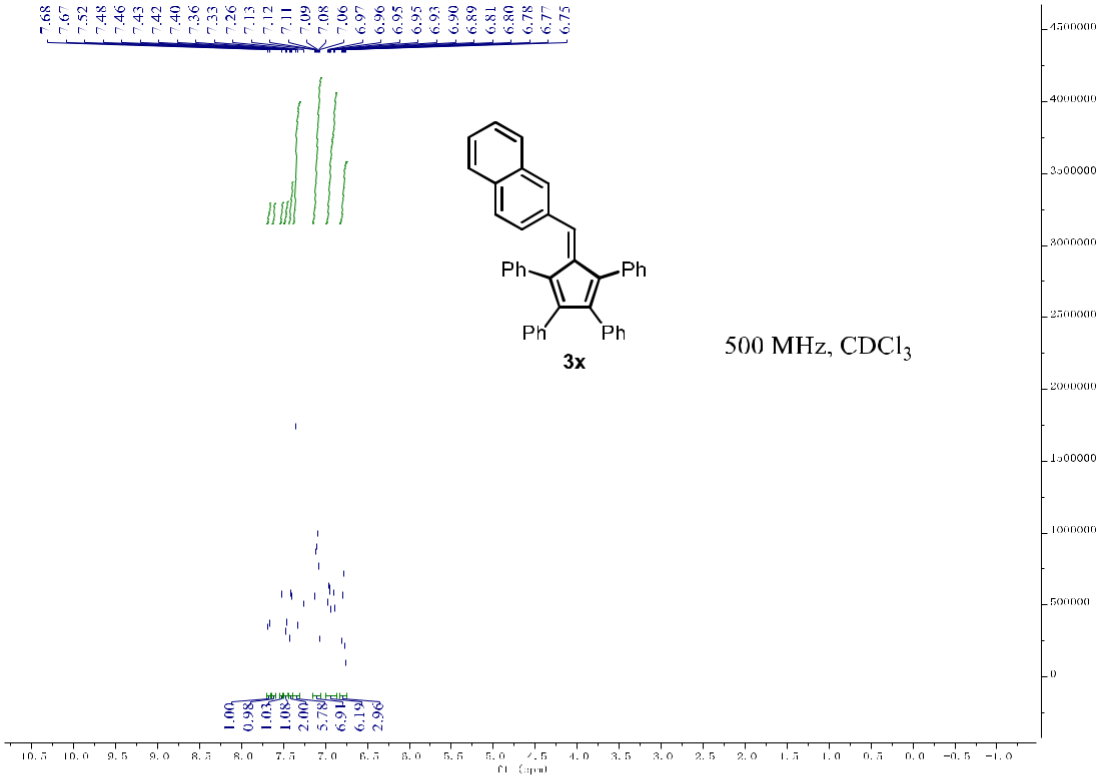


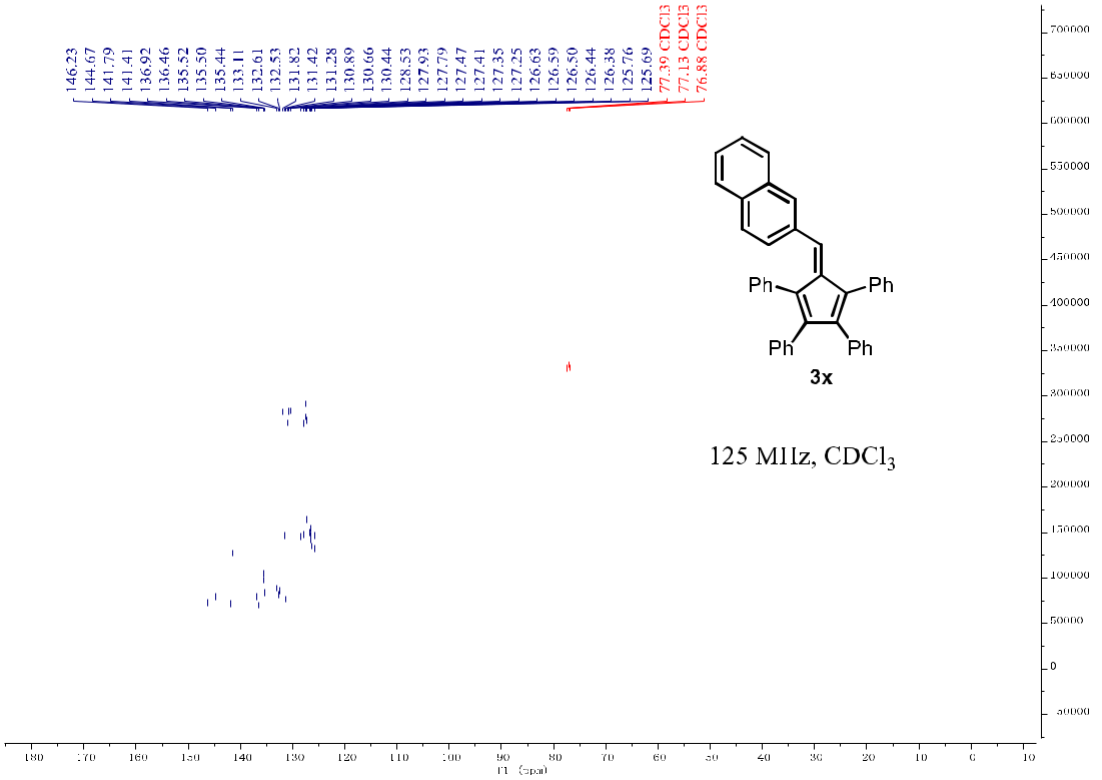
(5-(3,4-dimethoxybenzylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3w)



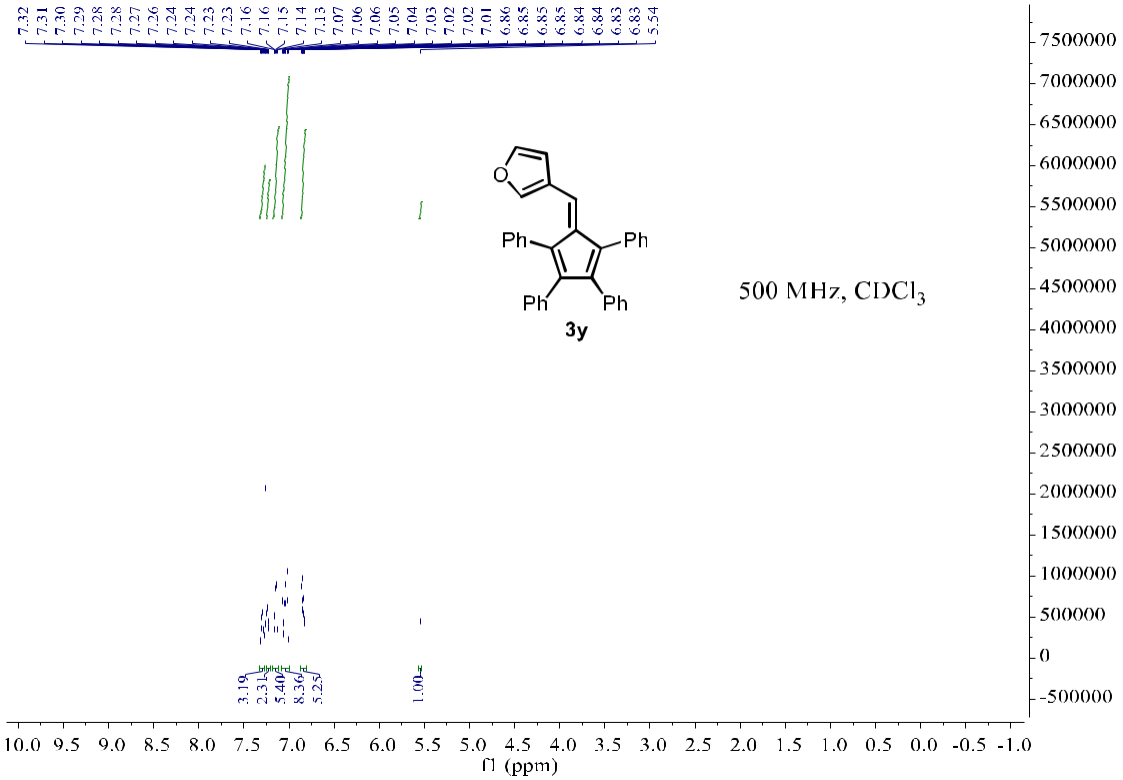


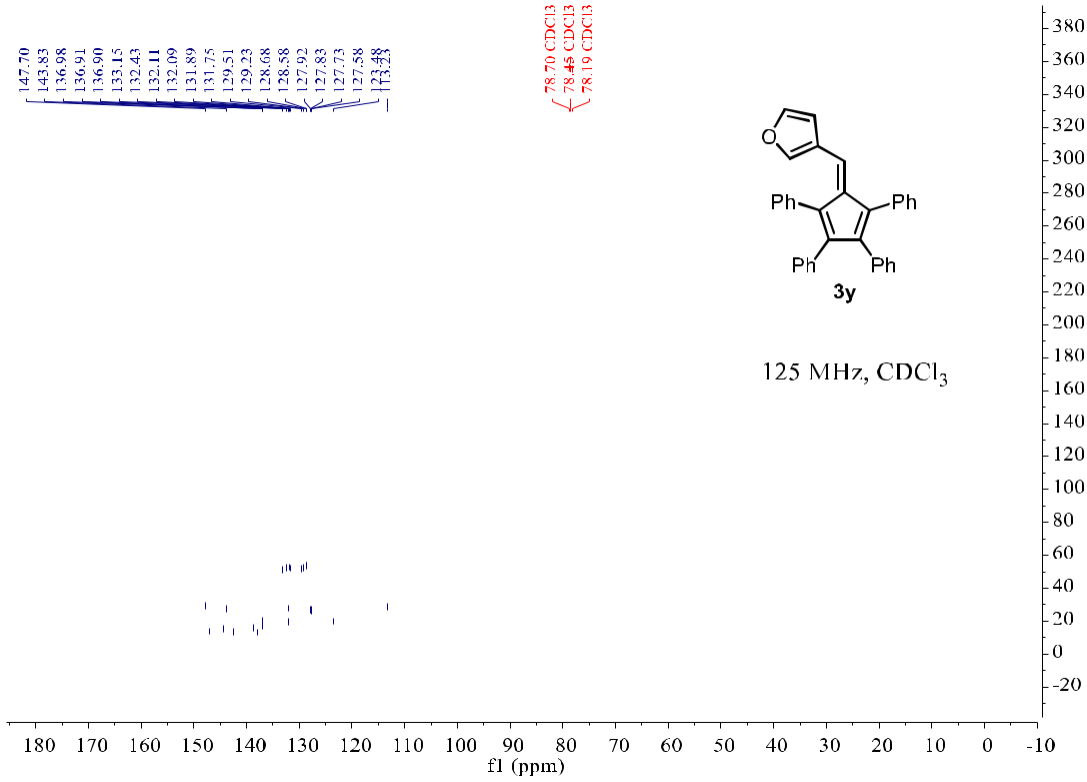
2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)naphthalene (3x)



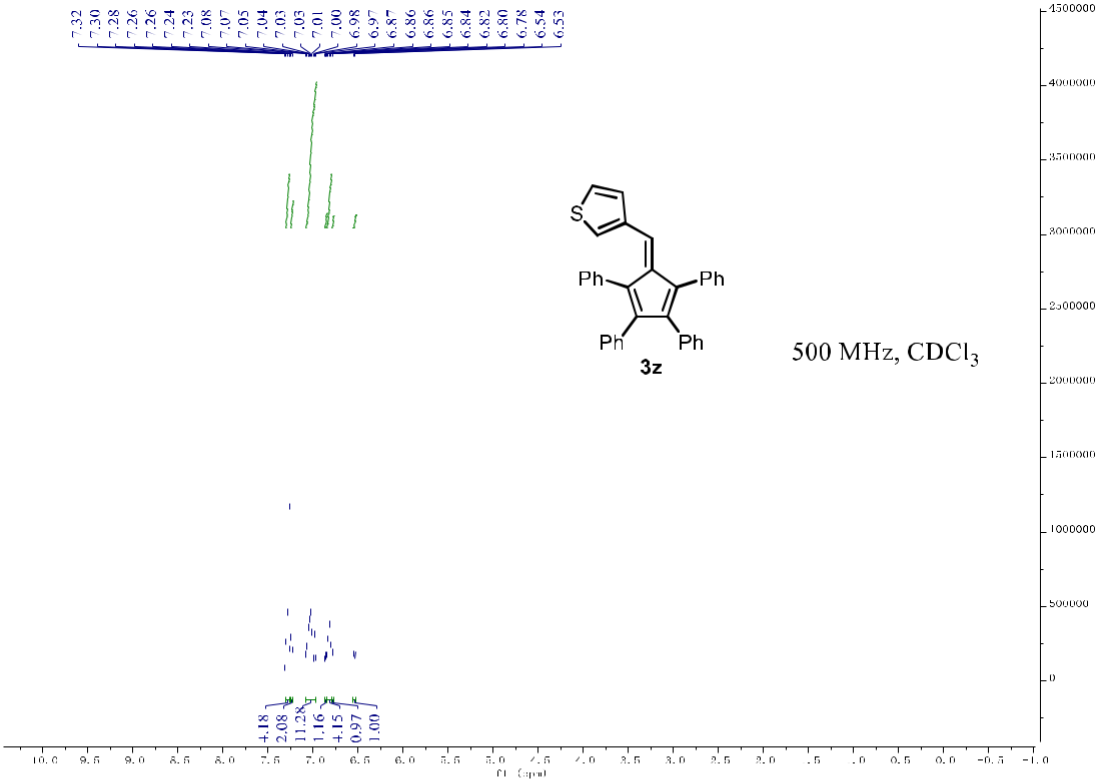


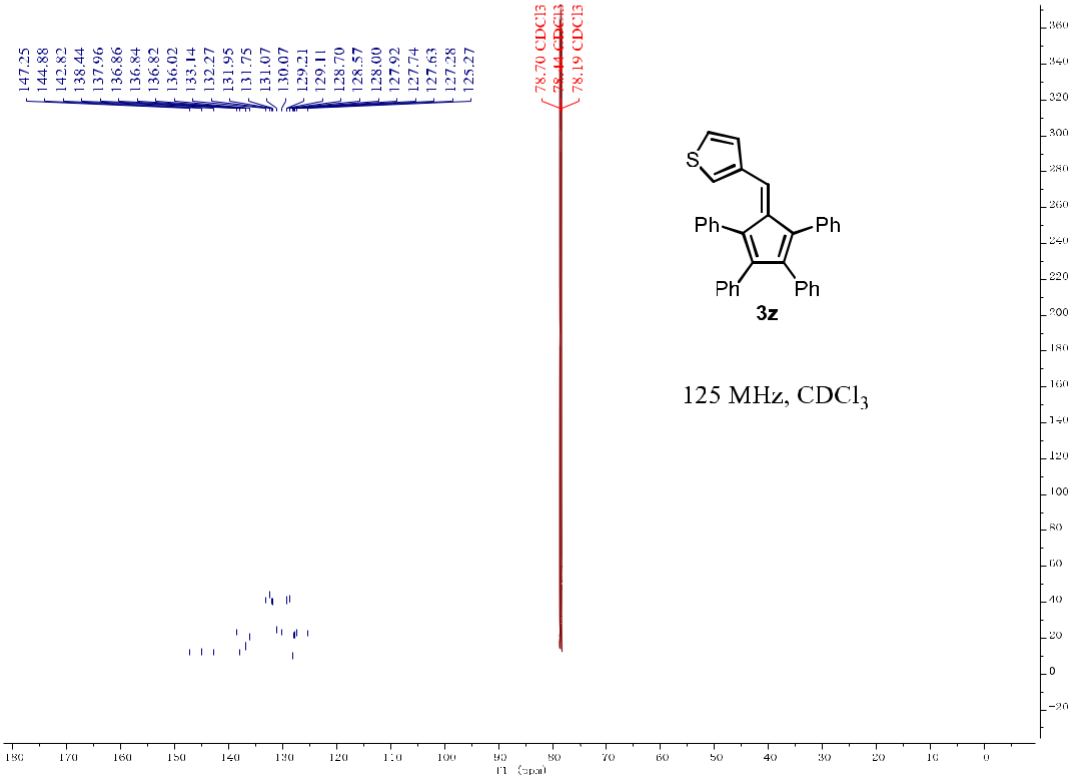
3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)furan (3y)



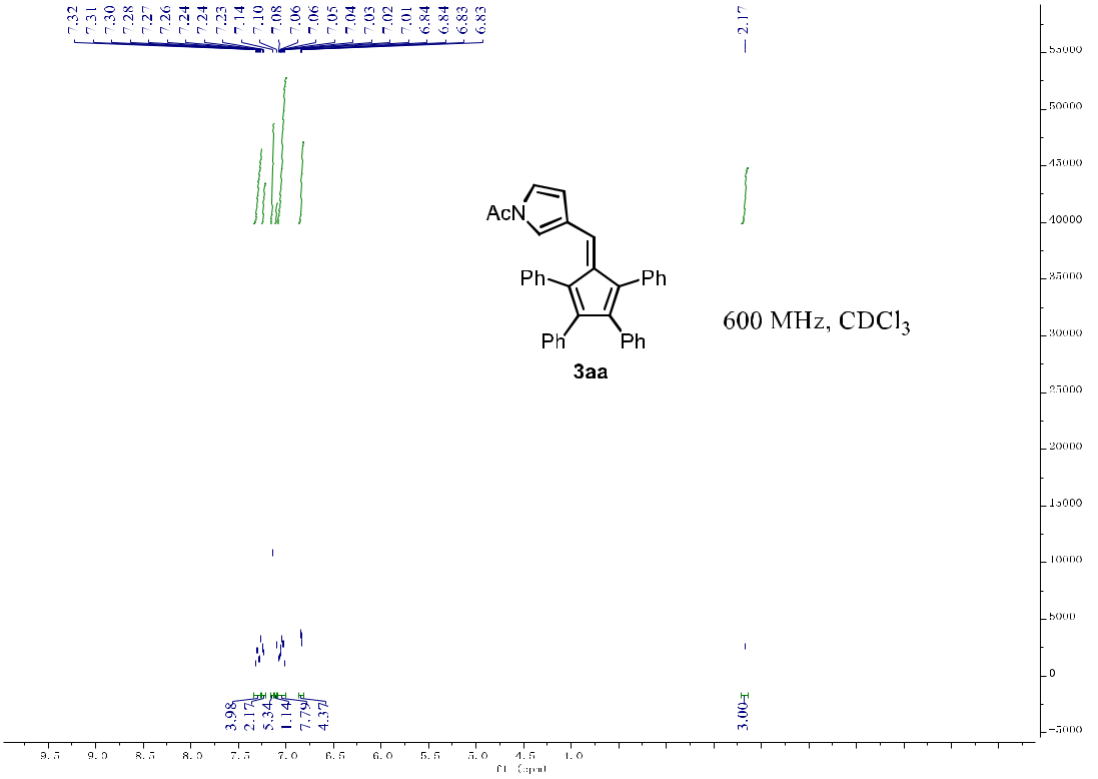


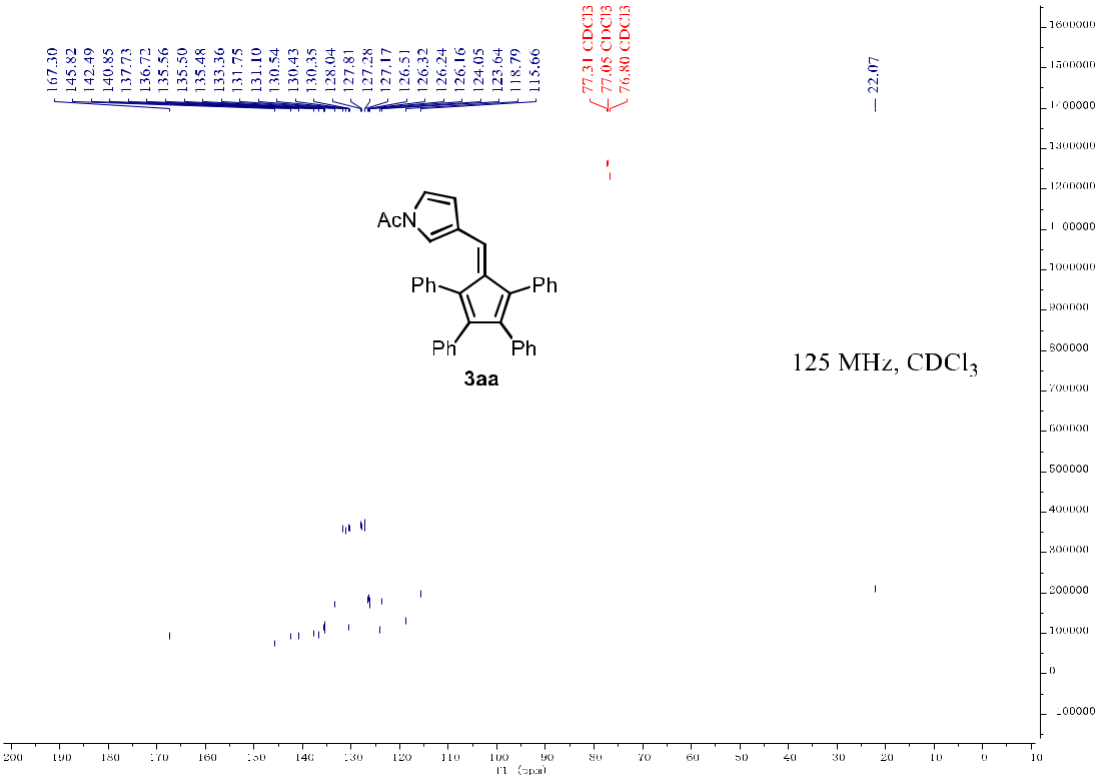
3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)thiophene (3z)



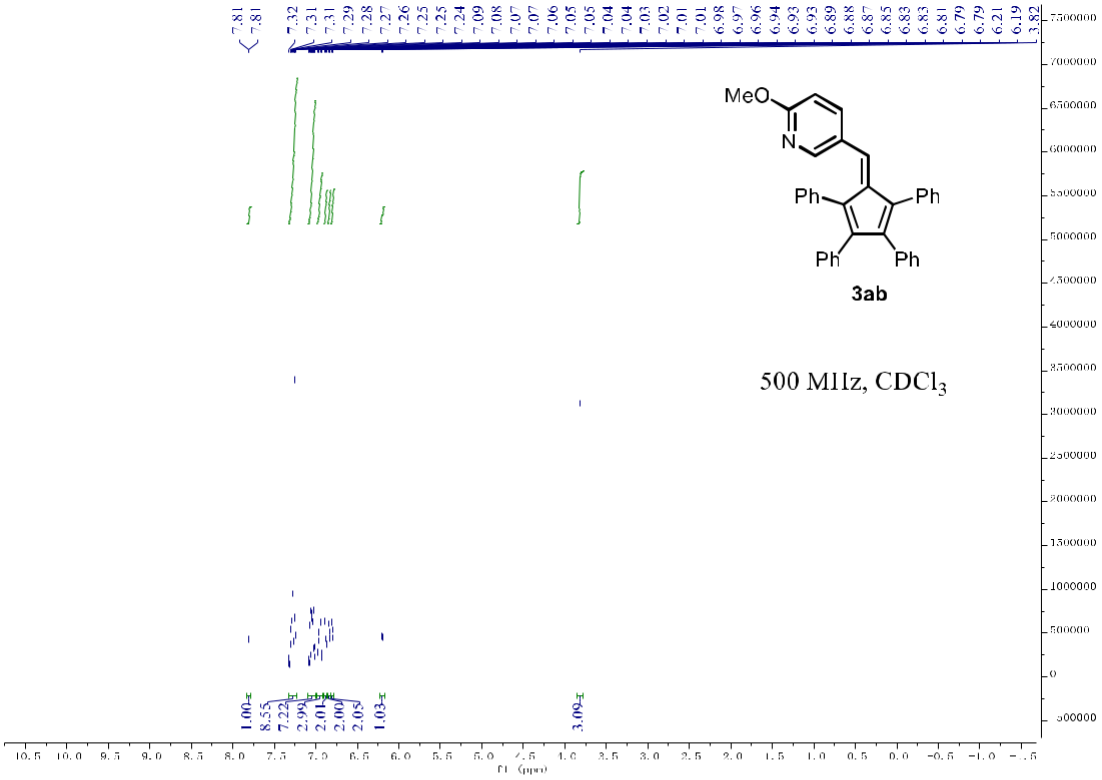


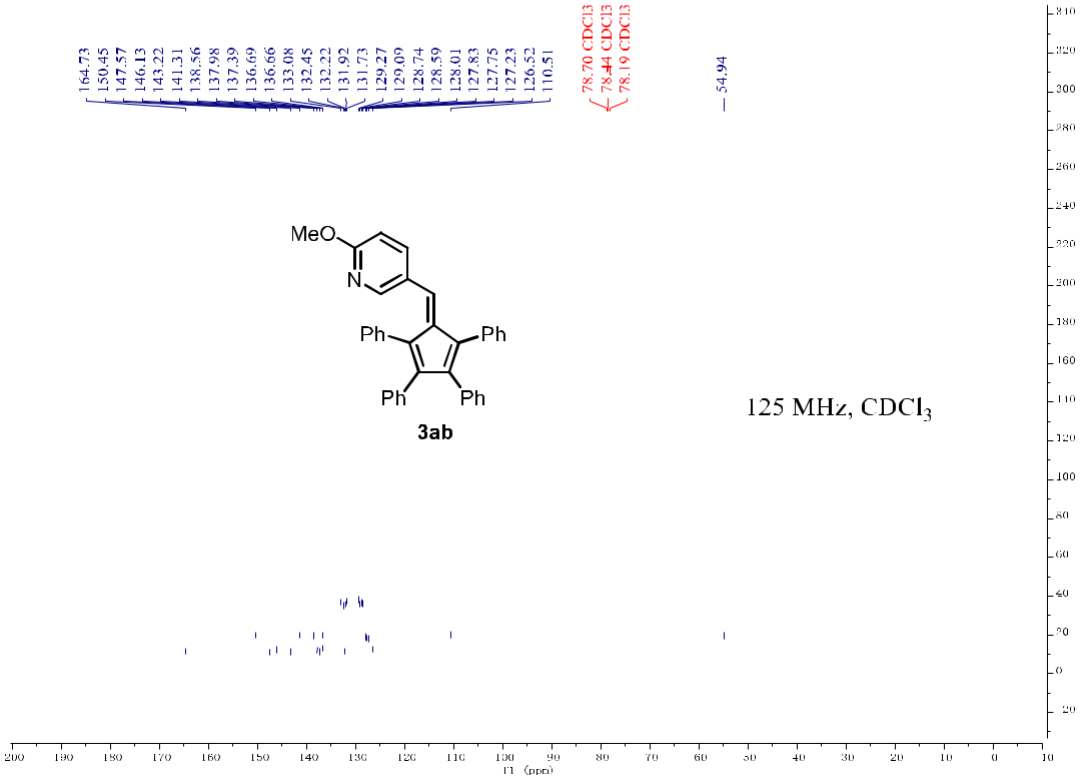
1-(3-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-1H-pyrrol-1-yl)ethan-1-one (3aa)



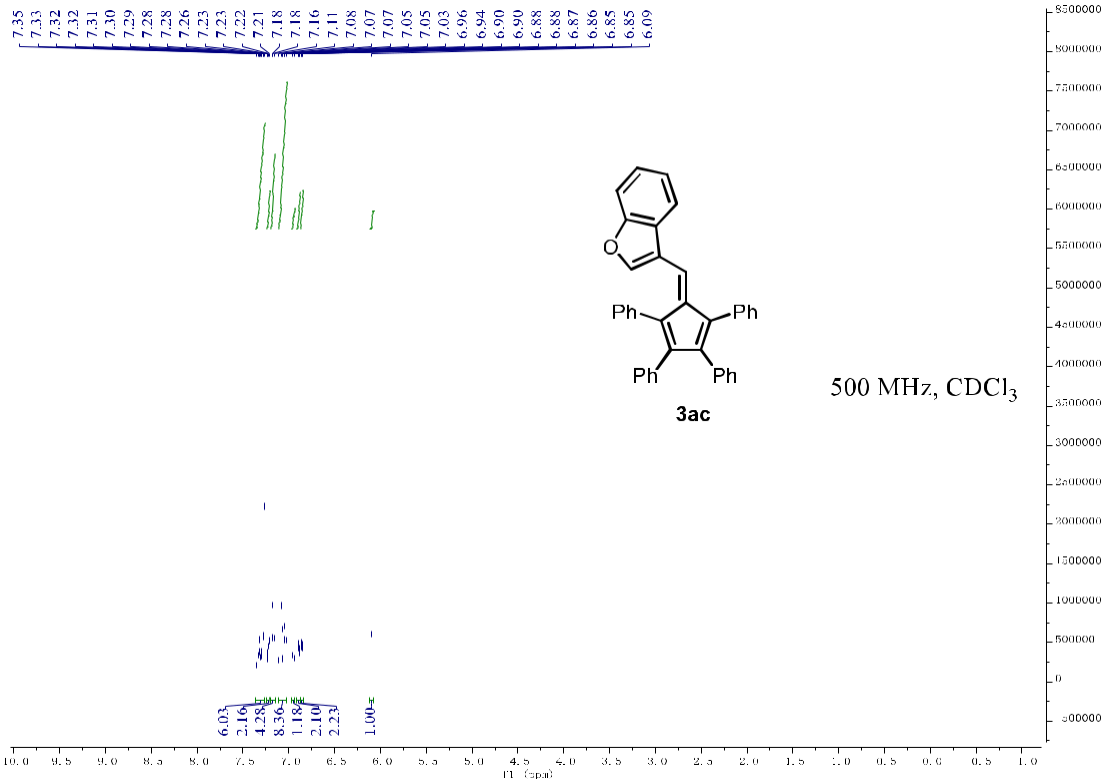


2-methoxy-5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)pyridine (3ab)

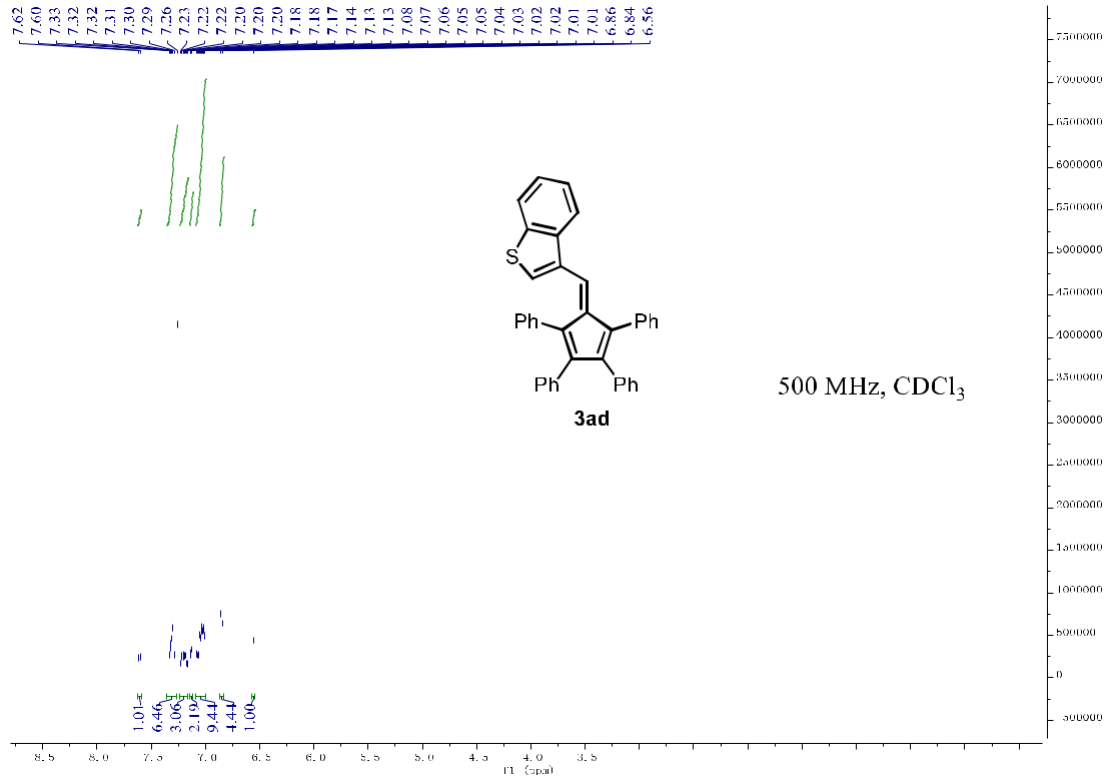


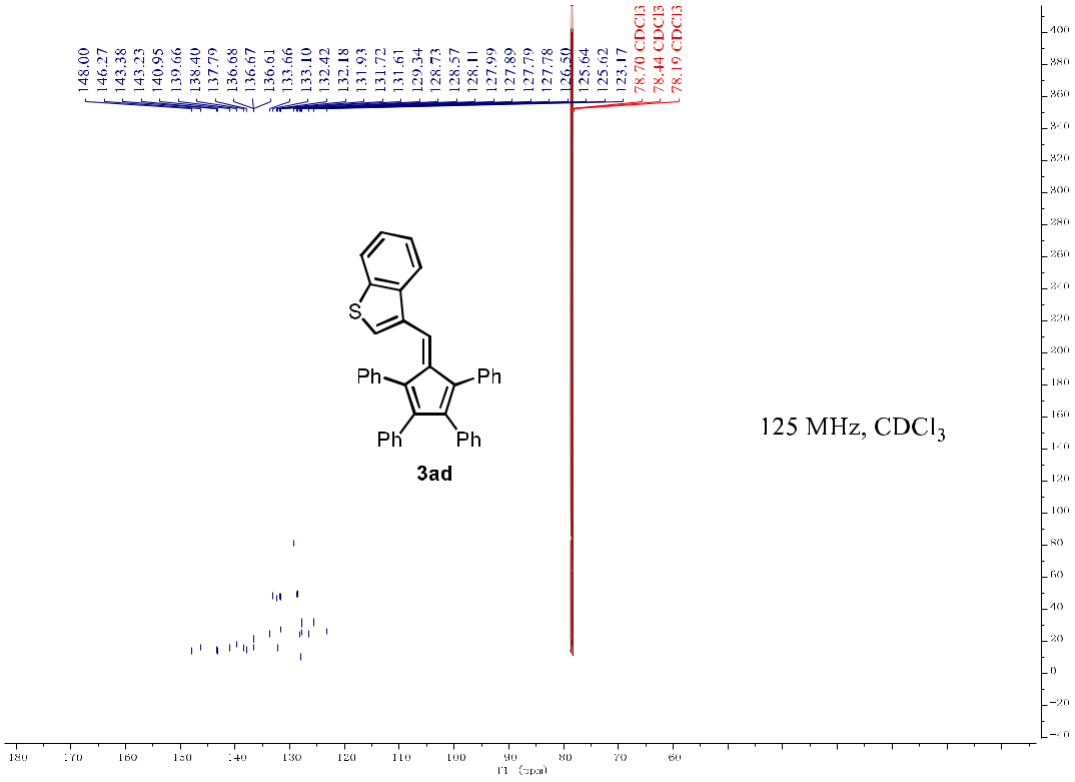


2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)benzofuran (3ac)

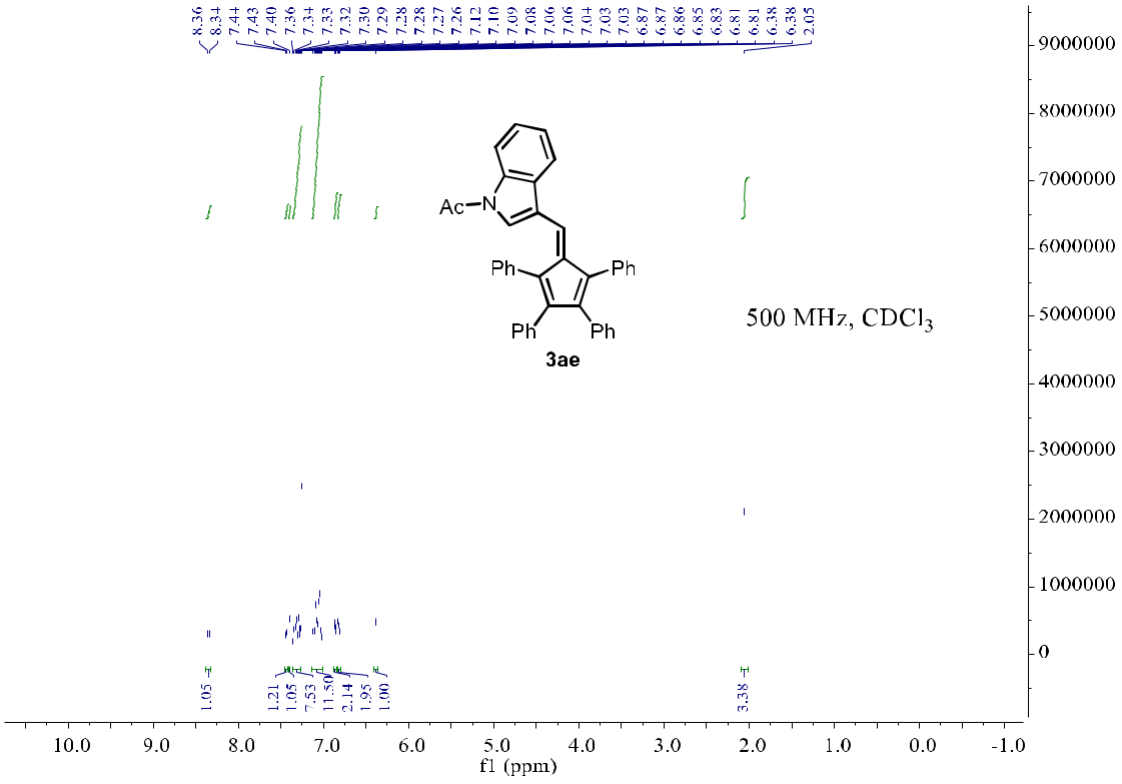


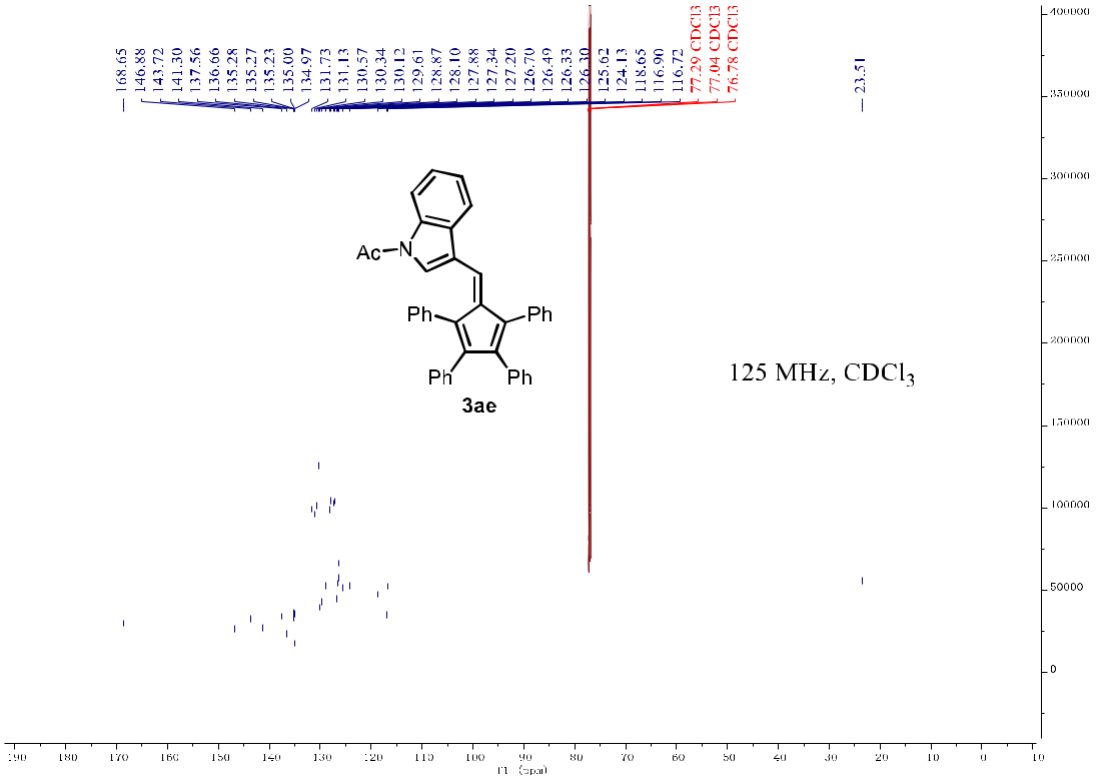
2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)benzo[b]thiophene (3ad)



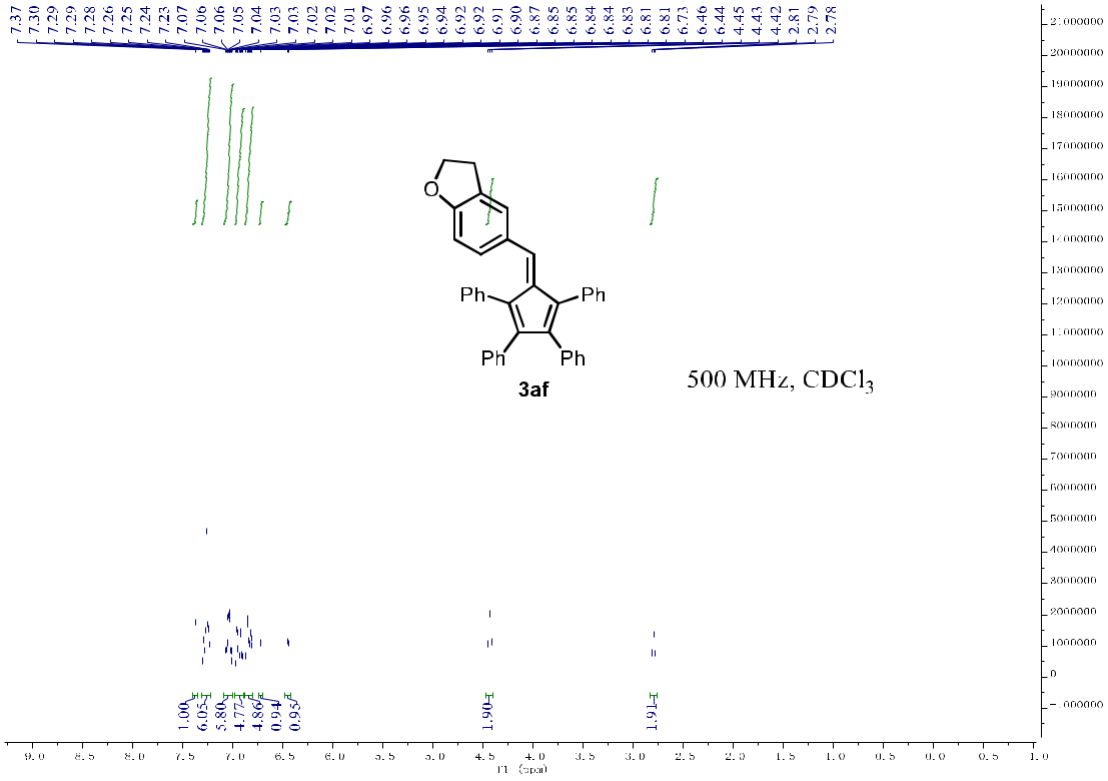


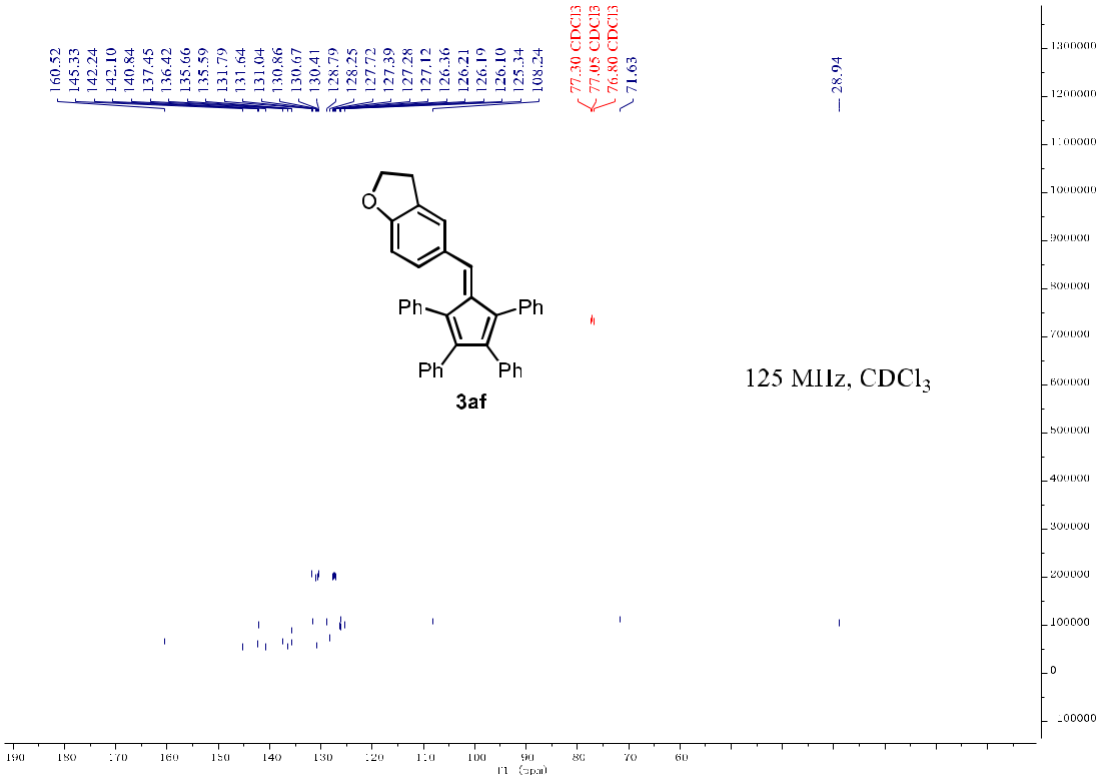
1-(2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-1H-indol-1-yl)ethan-1-one (3ae)



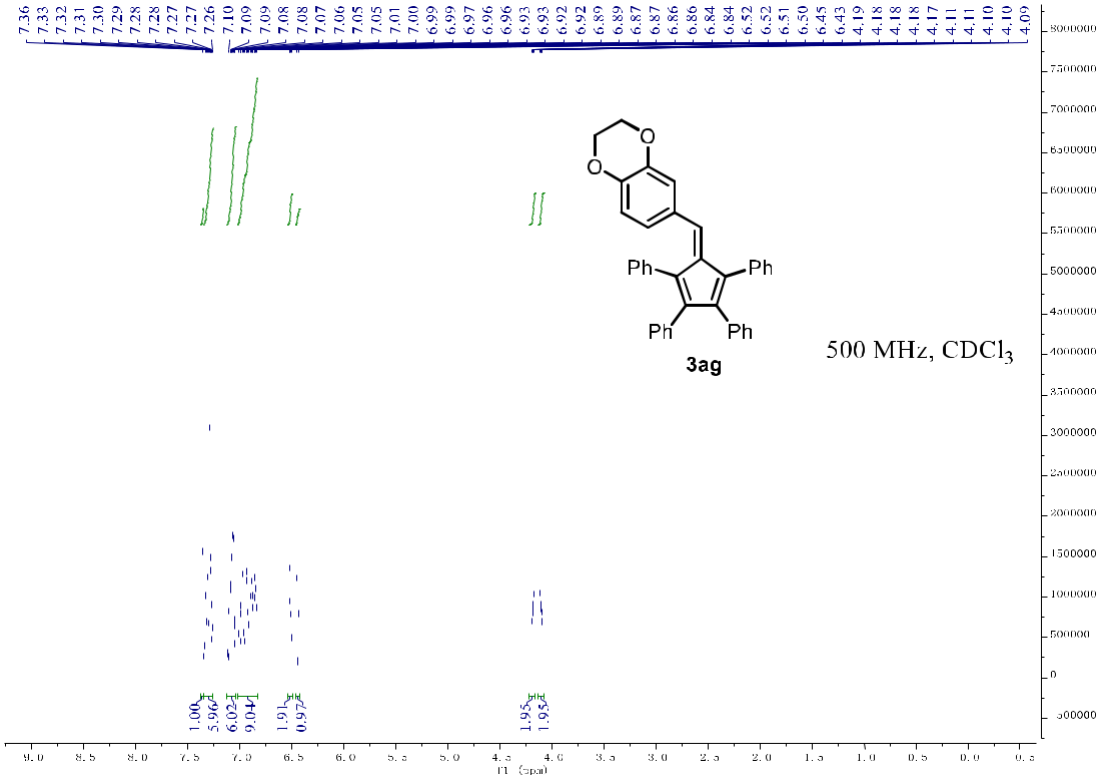


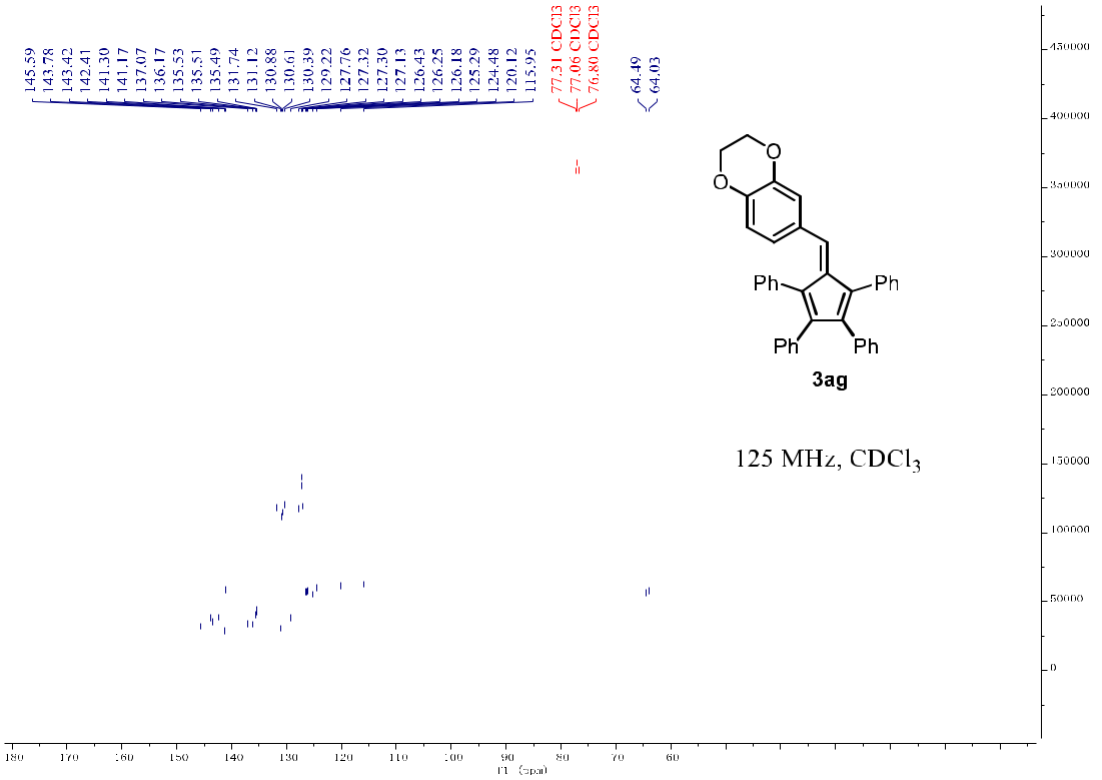
5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-2,3-dihydrobenzofuran (3af)



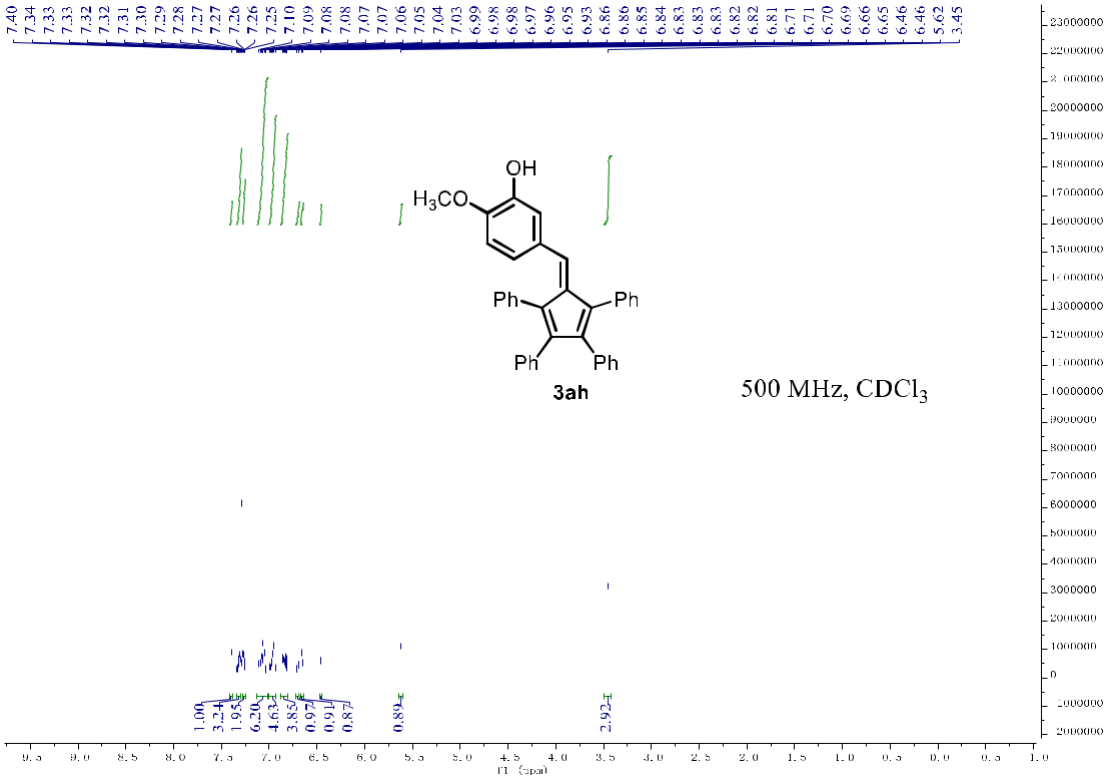


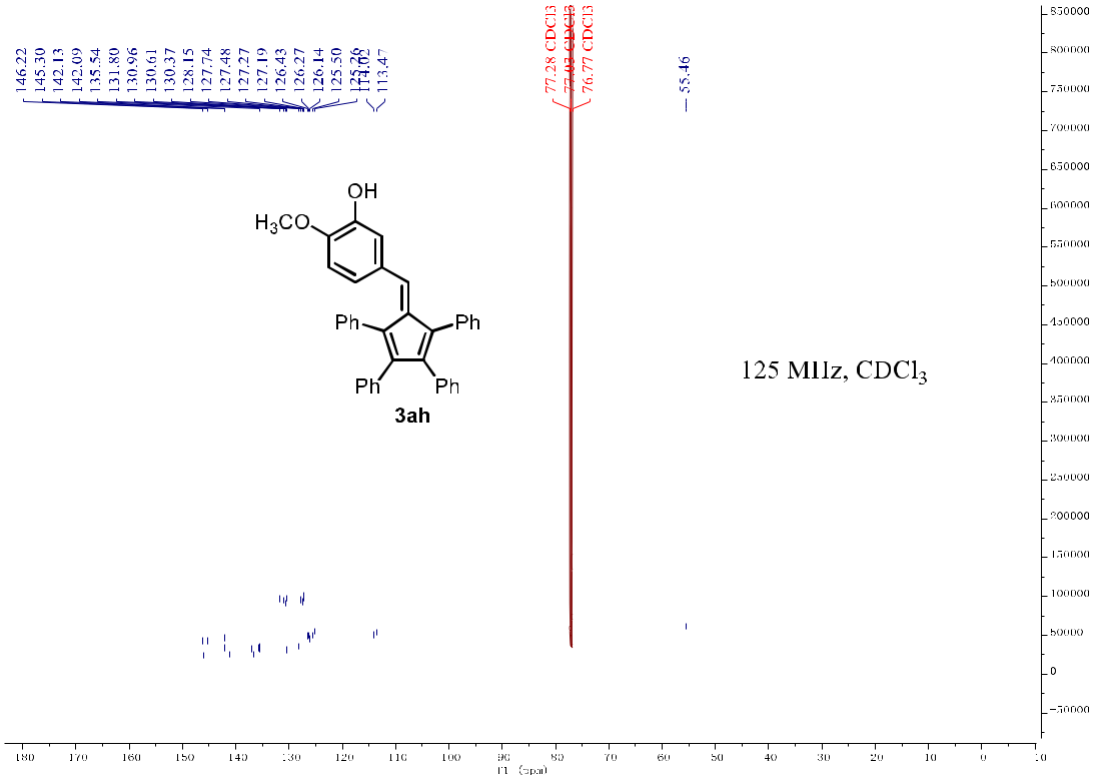
6-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)-2,3-dihydrobenzo[b][1,4]dioxine (3ag)



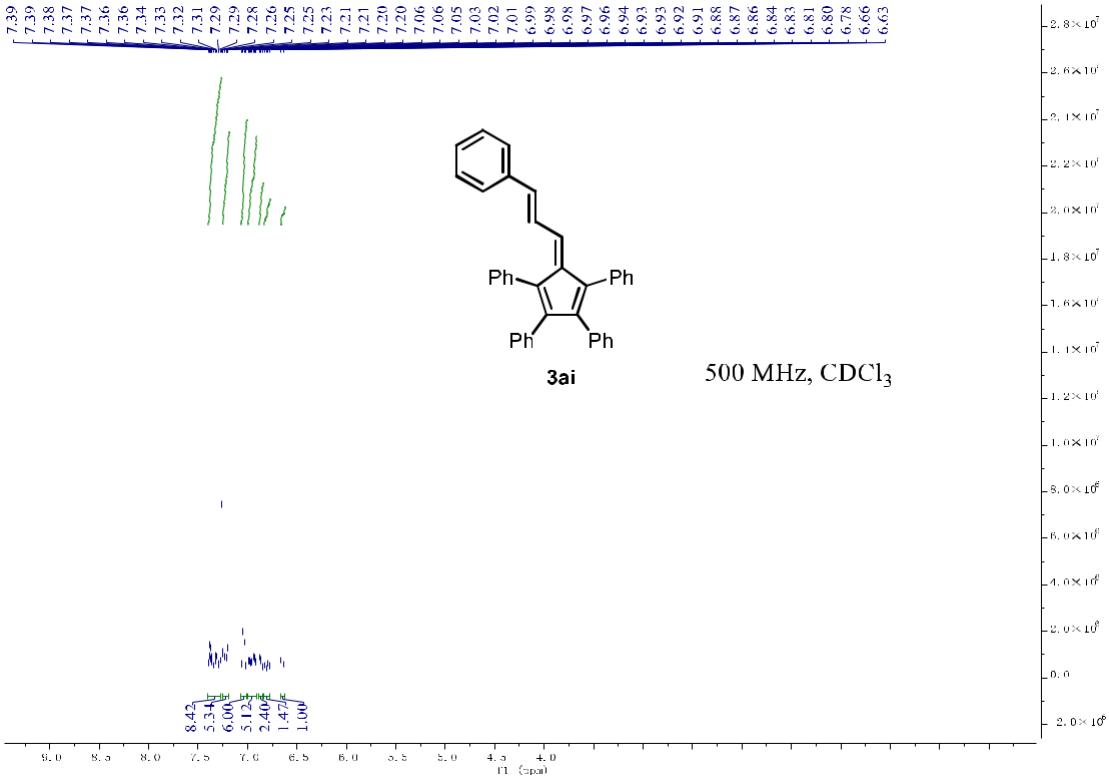


2-methoxy-5-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenol (3ah)

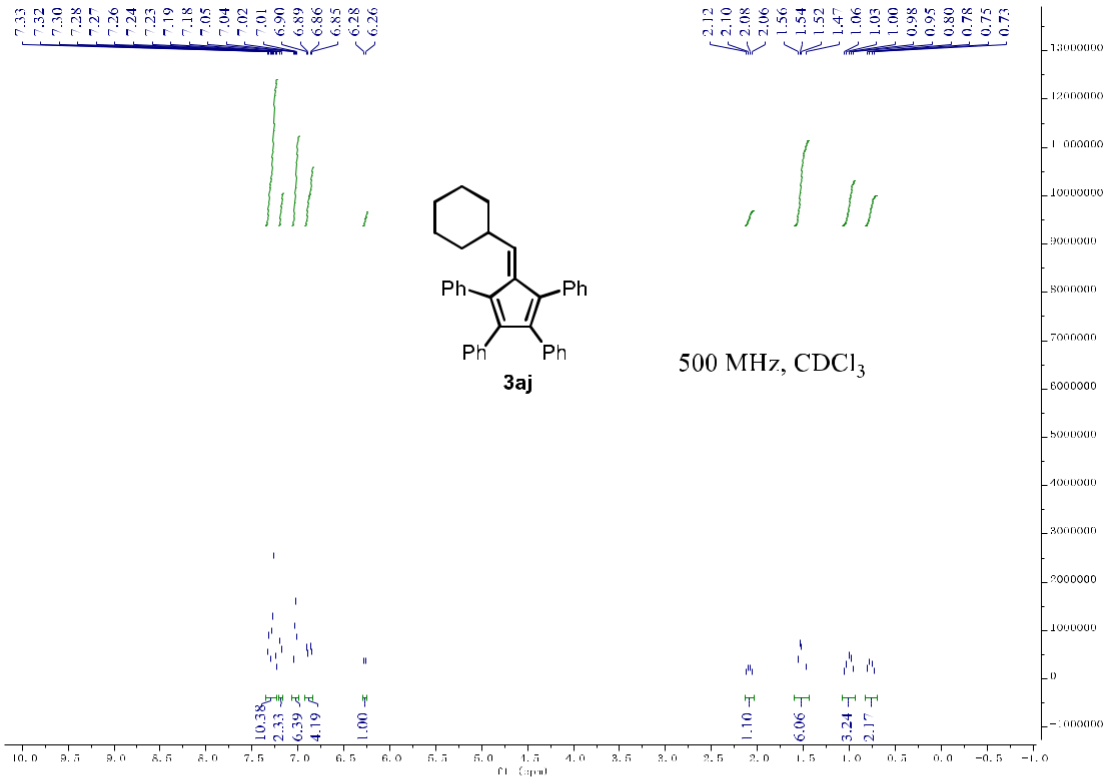


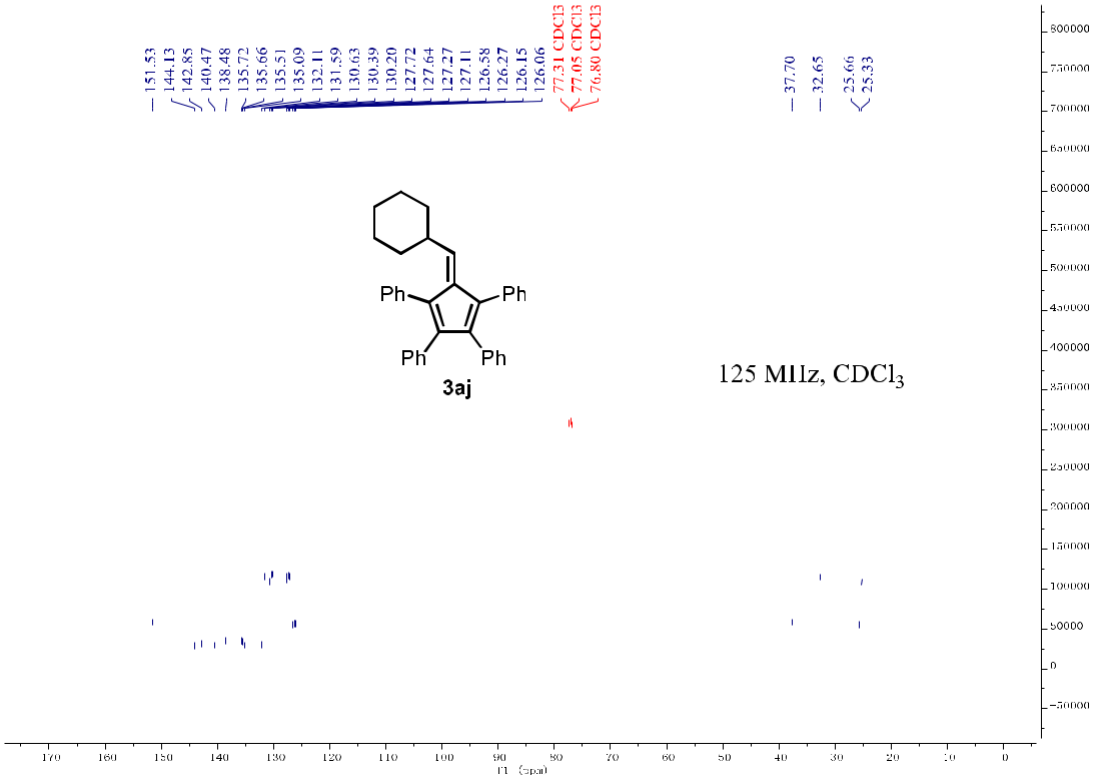


(E)-(5-(3-phenylallylidene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3ai)

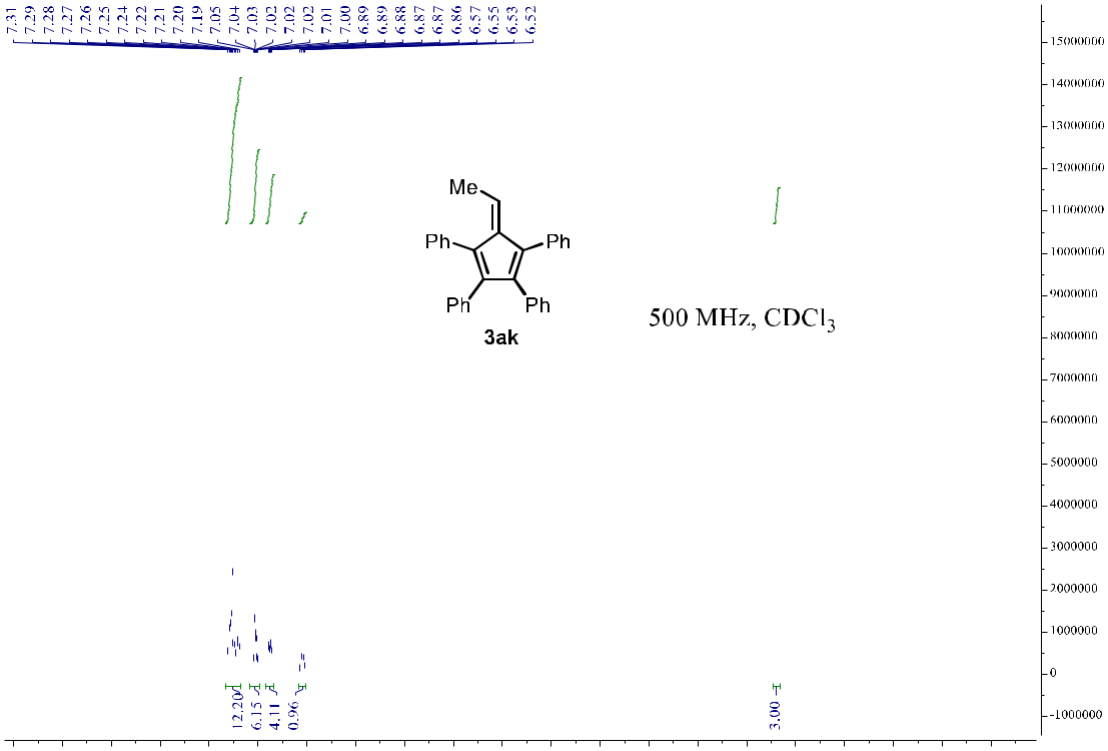


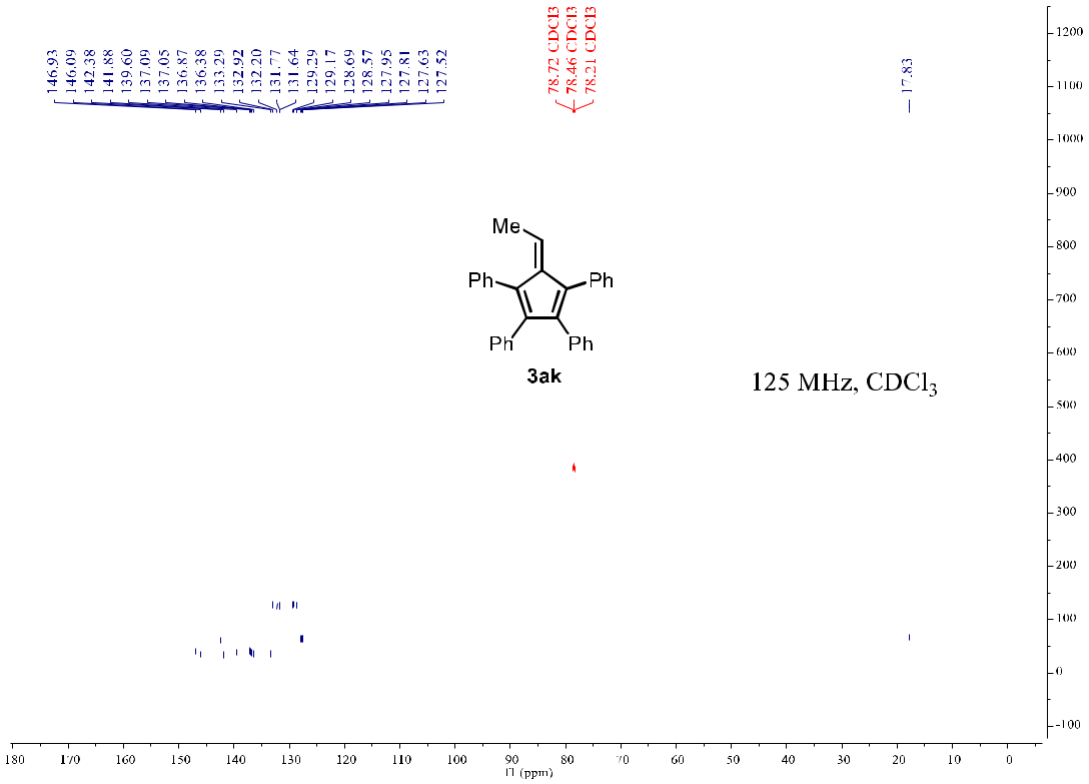
(5-(cyclohexylmethylene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3aj)



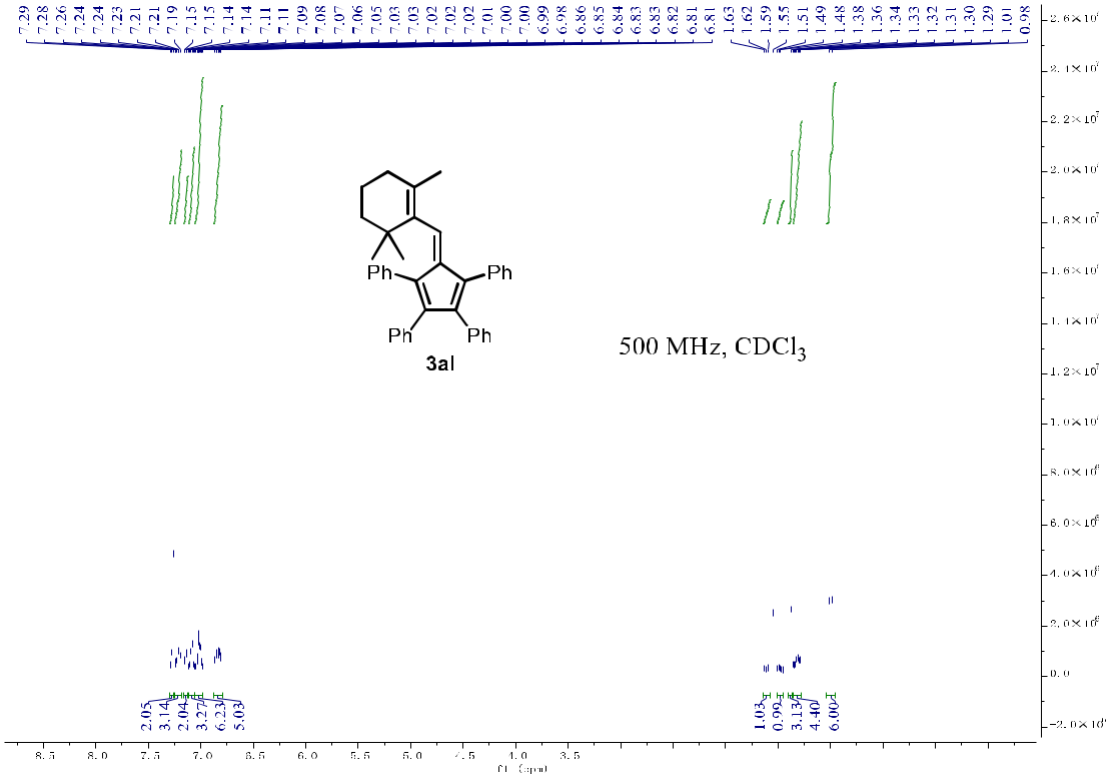


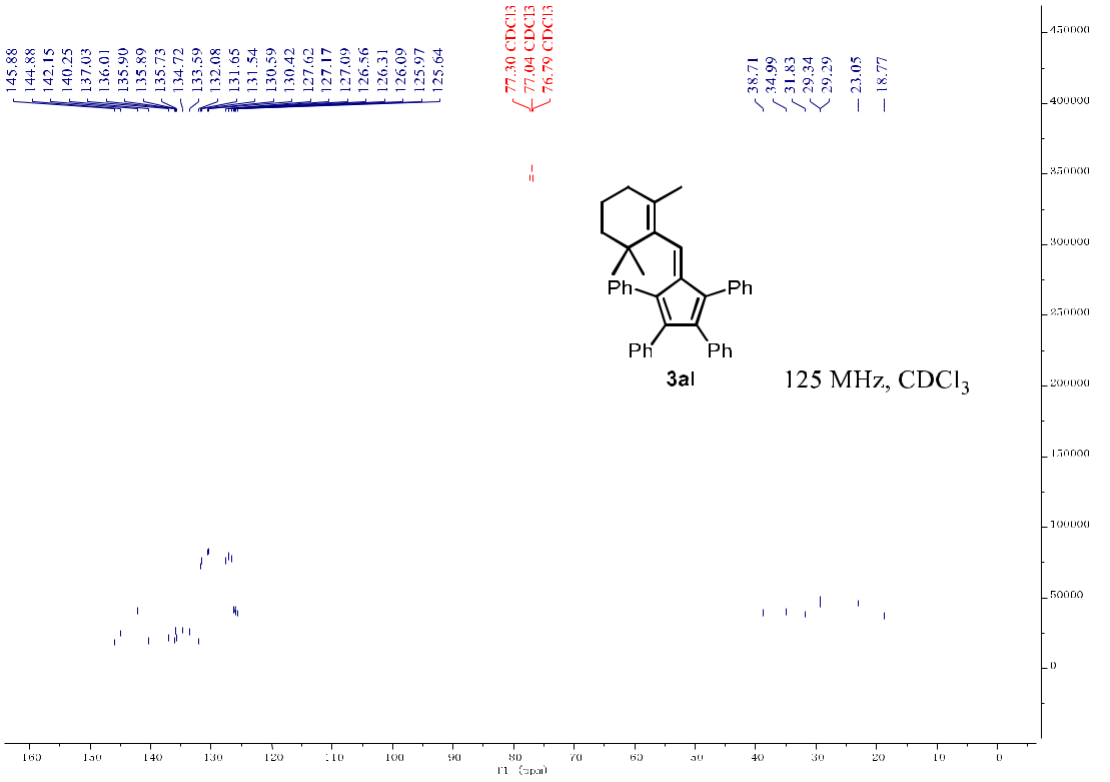
(5-ethylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3ak)



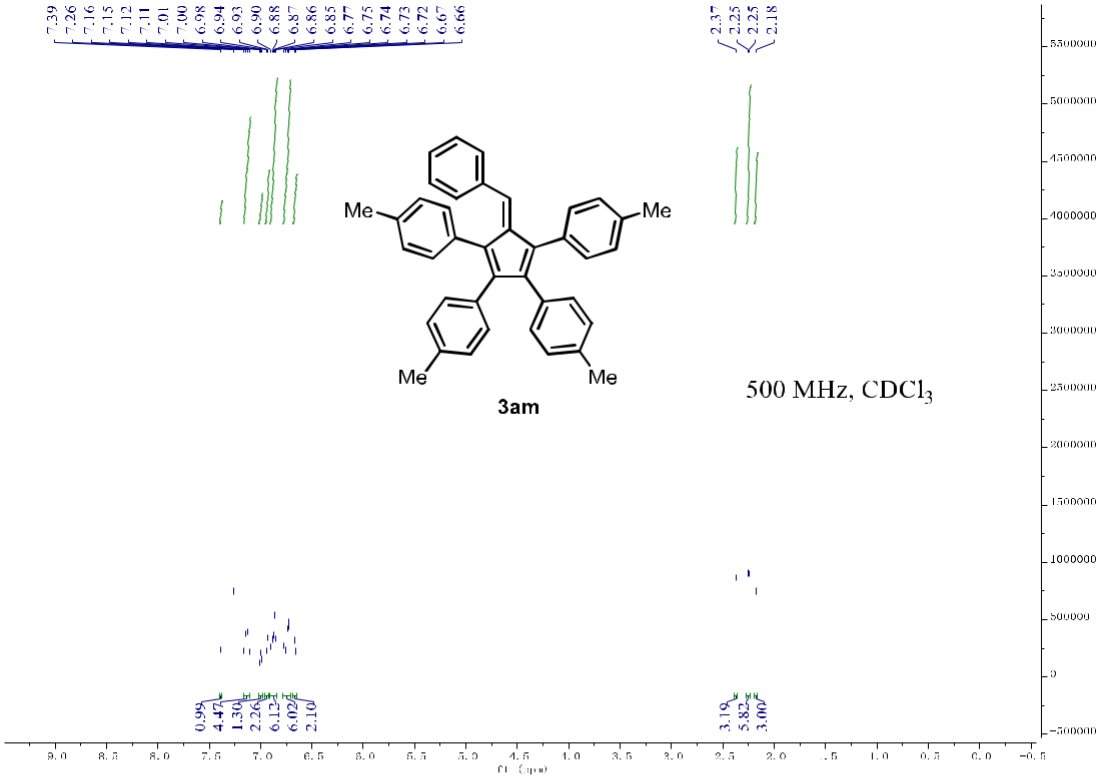


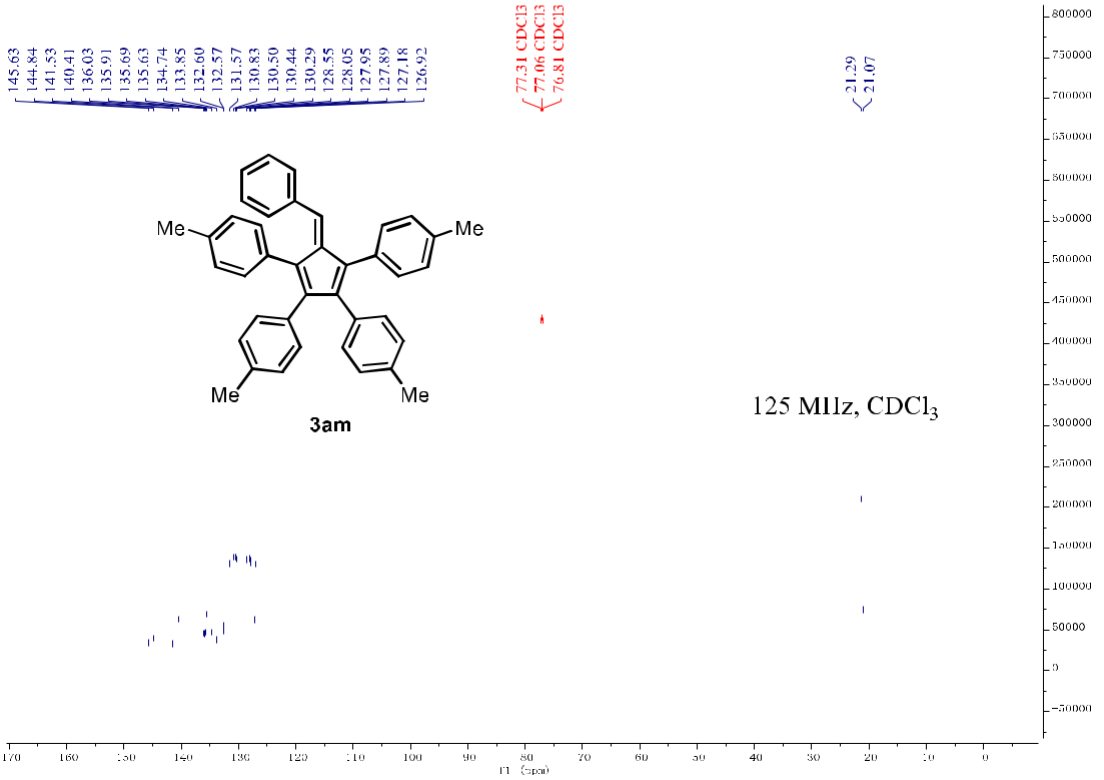
(5-((2,6,6-trimethylcyclohex-1-en-1-yl)methylene)cyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrabenzene (3al)



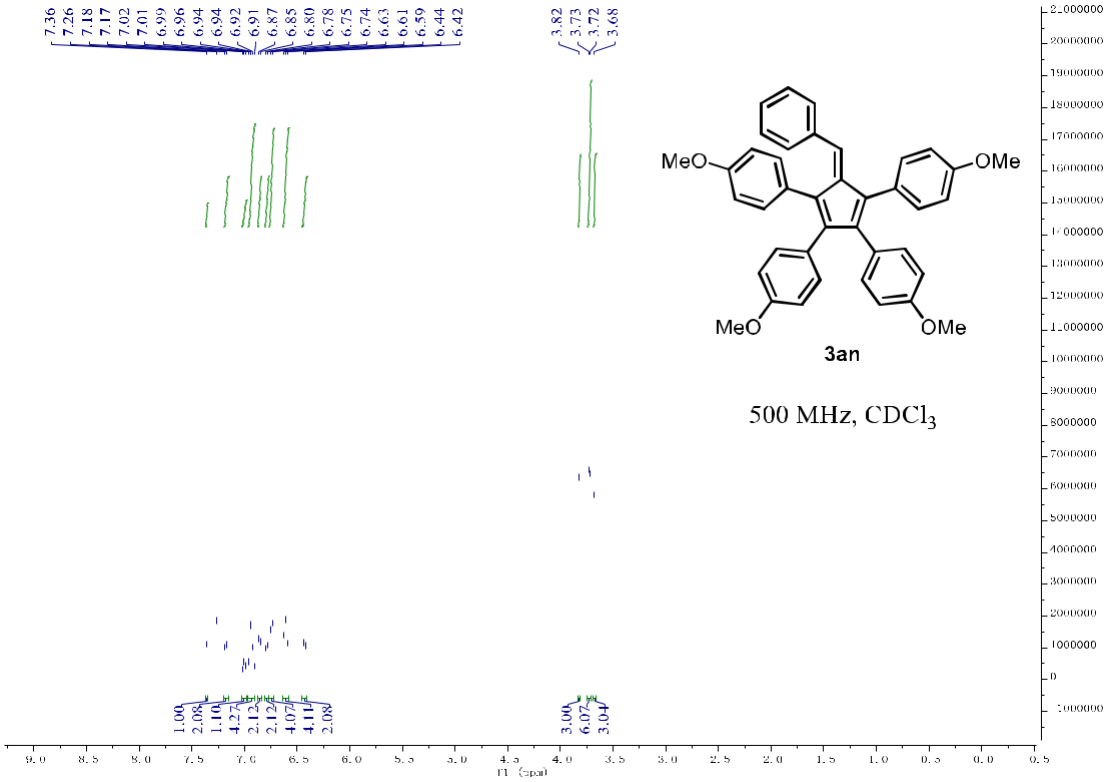


4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methylbenzene) (3am)

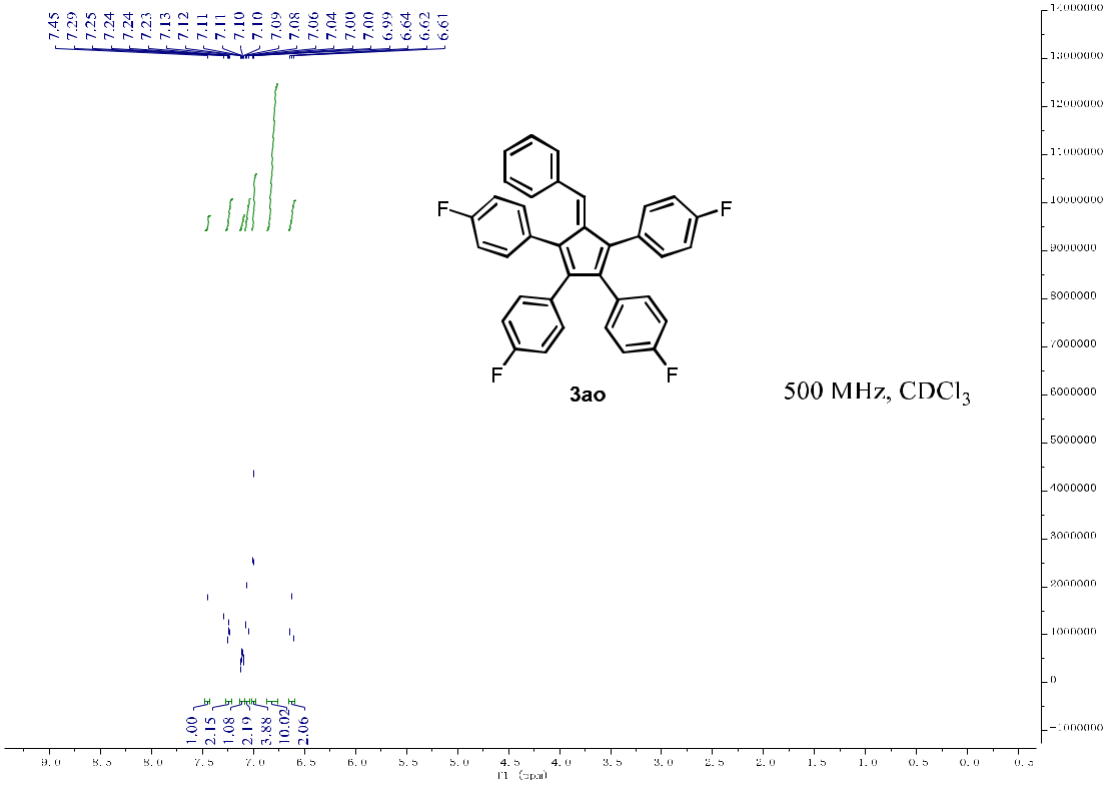


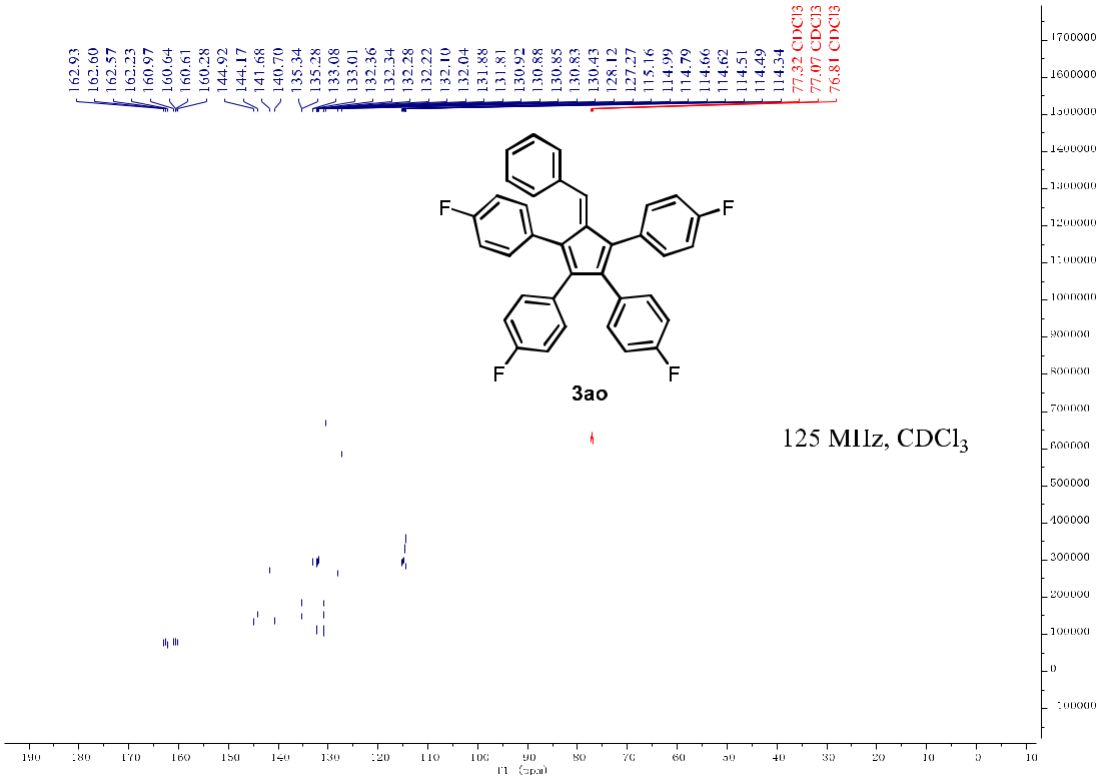


4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methoxybenzene) (3an)

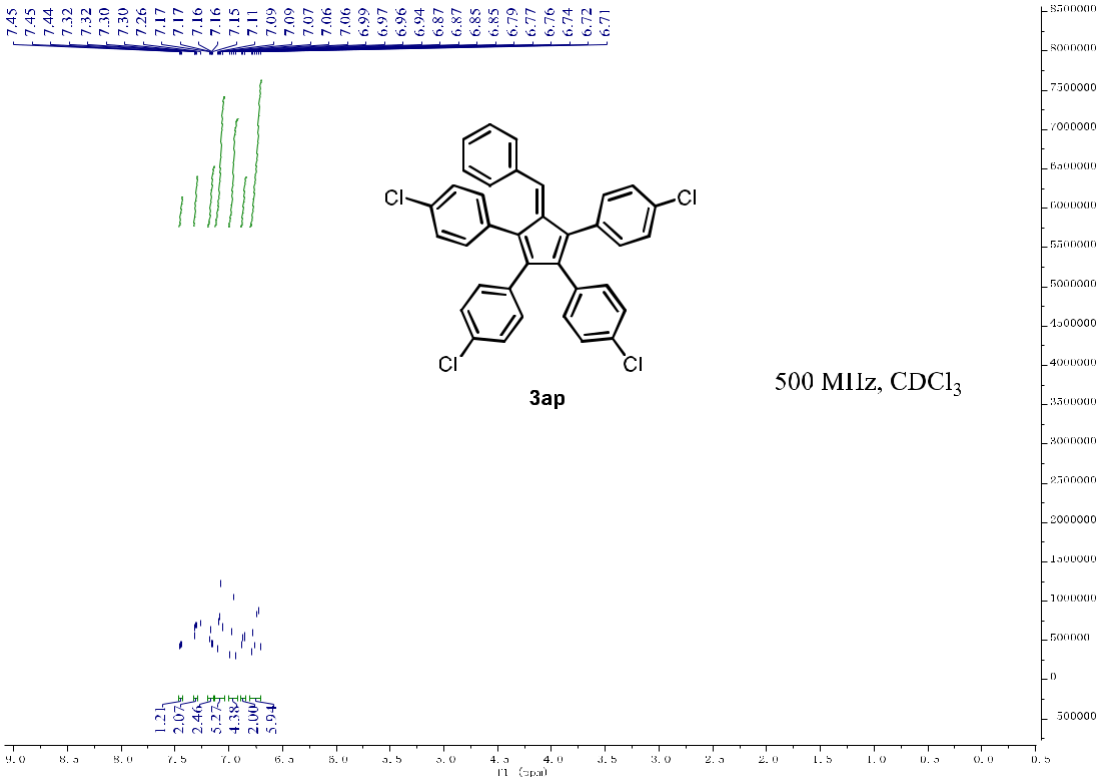


4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene) (3ao)

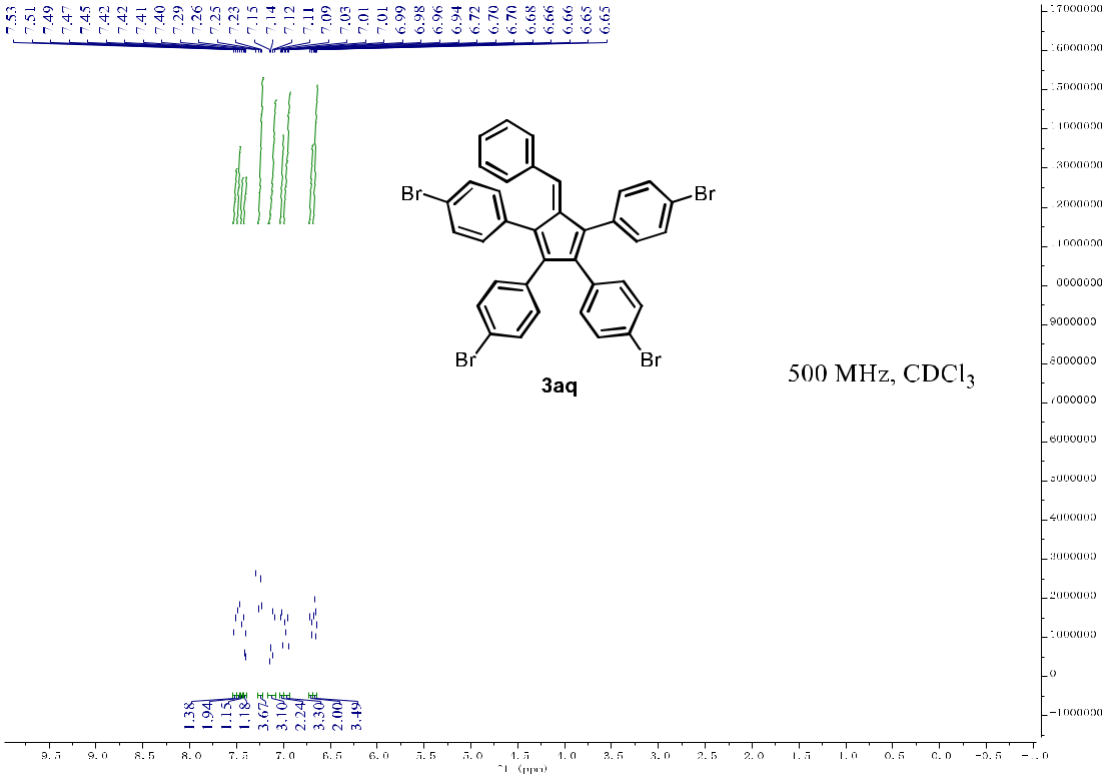


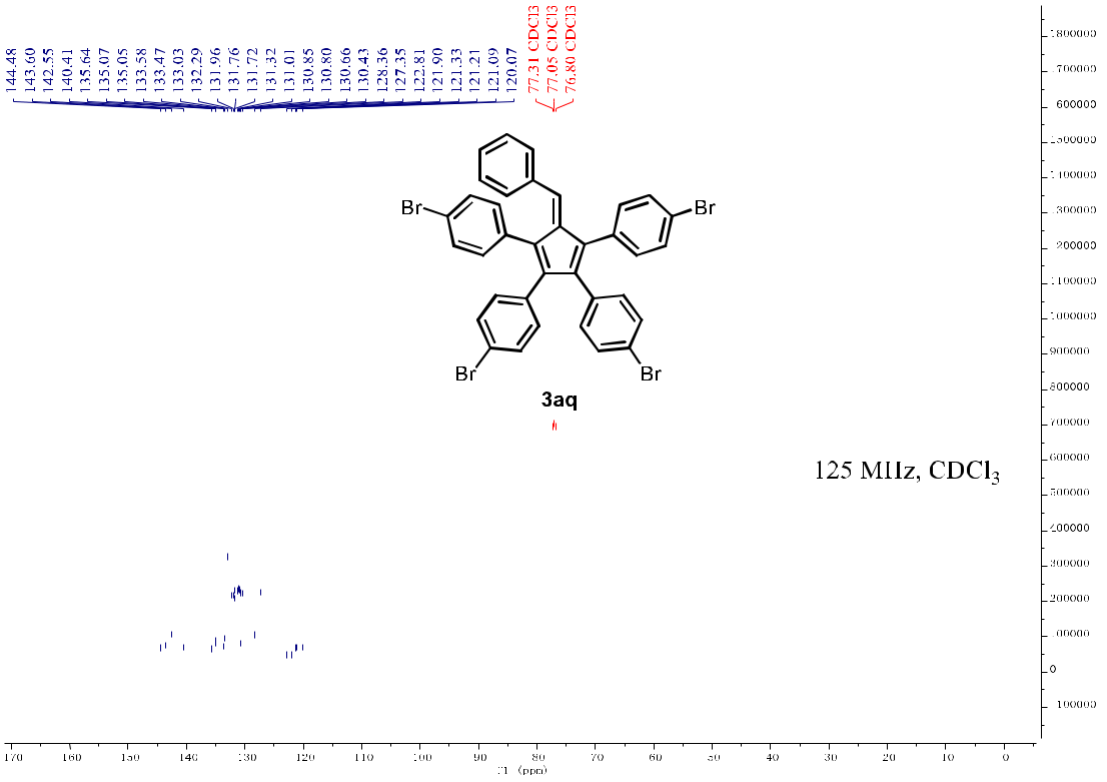


4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(chlorobenzene) (3ap)

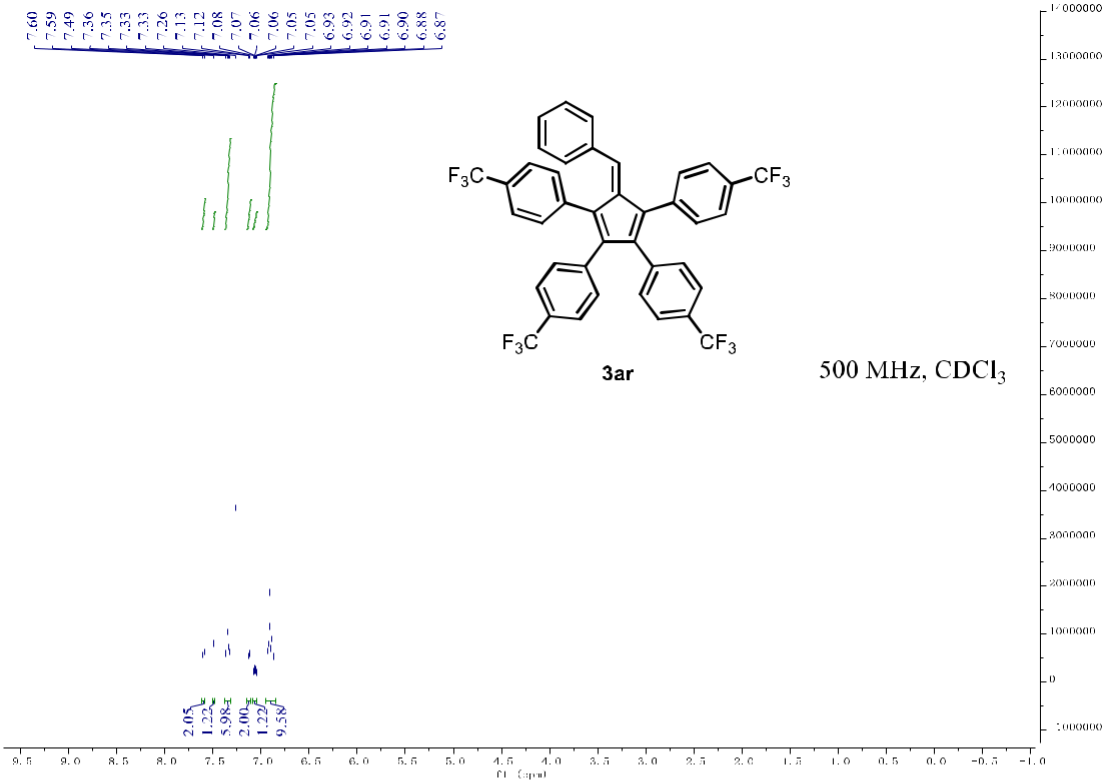


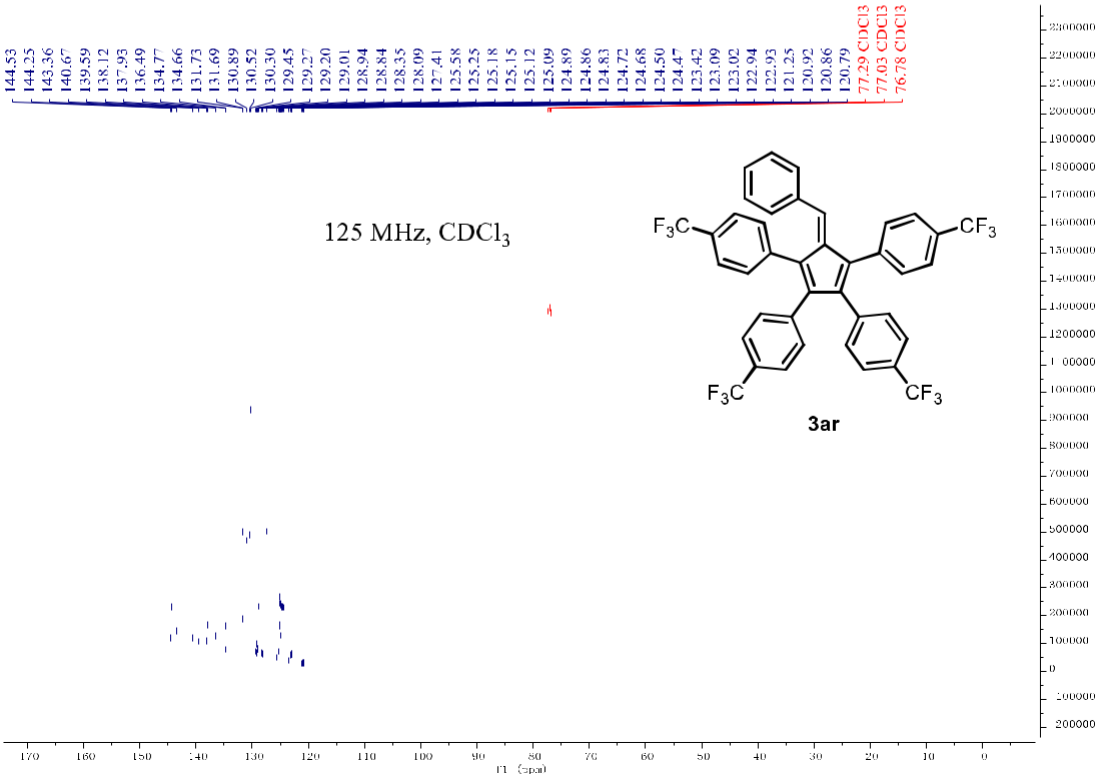
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(bromobenzene) (3aq)



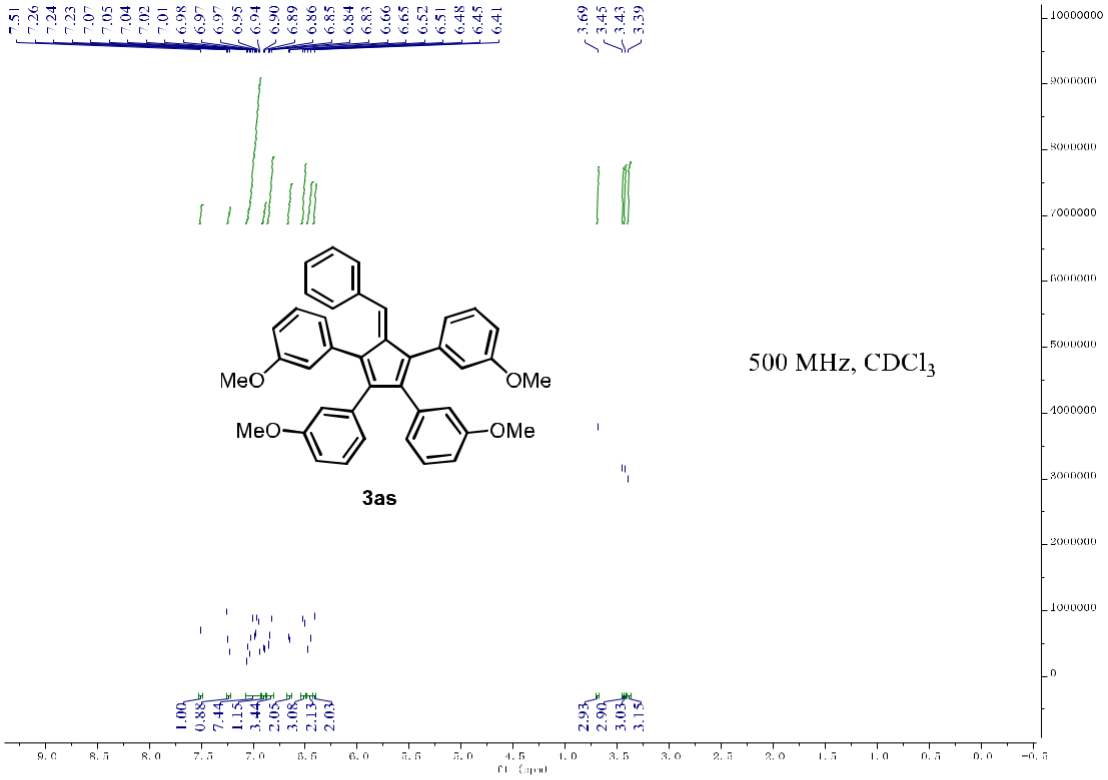


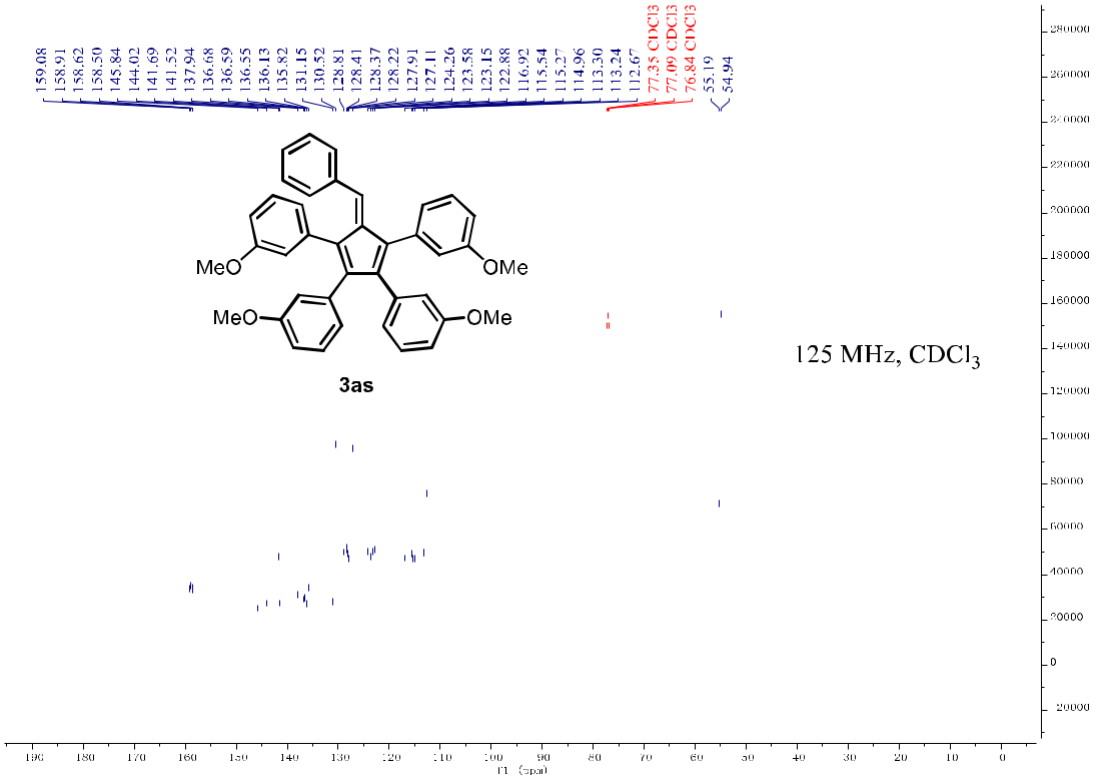
4,4',4'',4'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis((trifluoromethyl)benzene) (3ar)



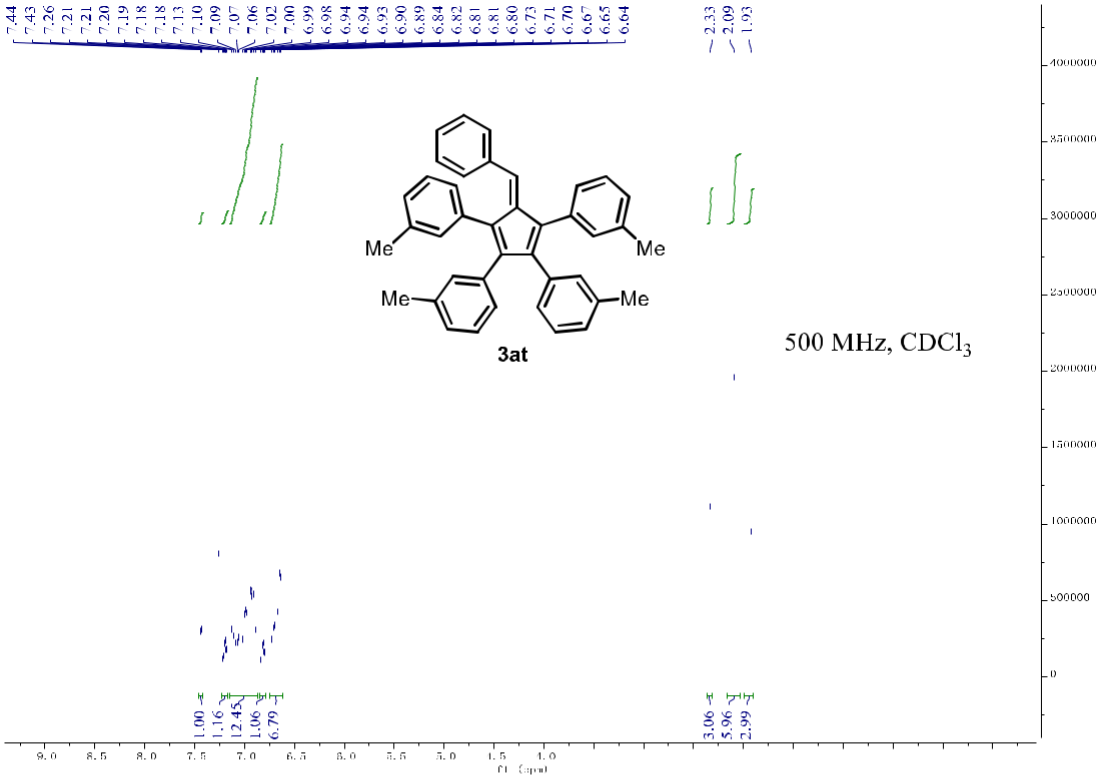


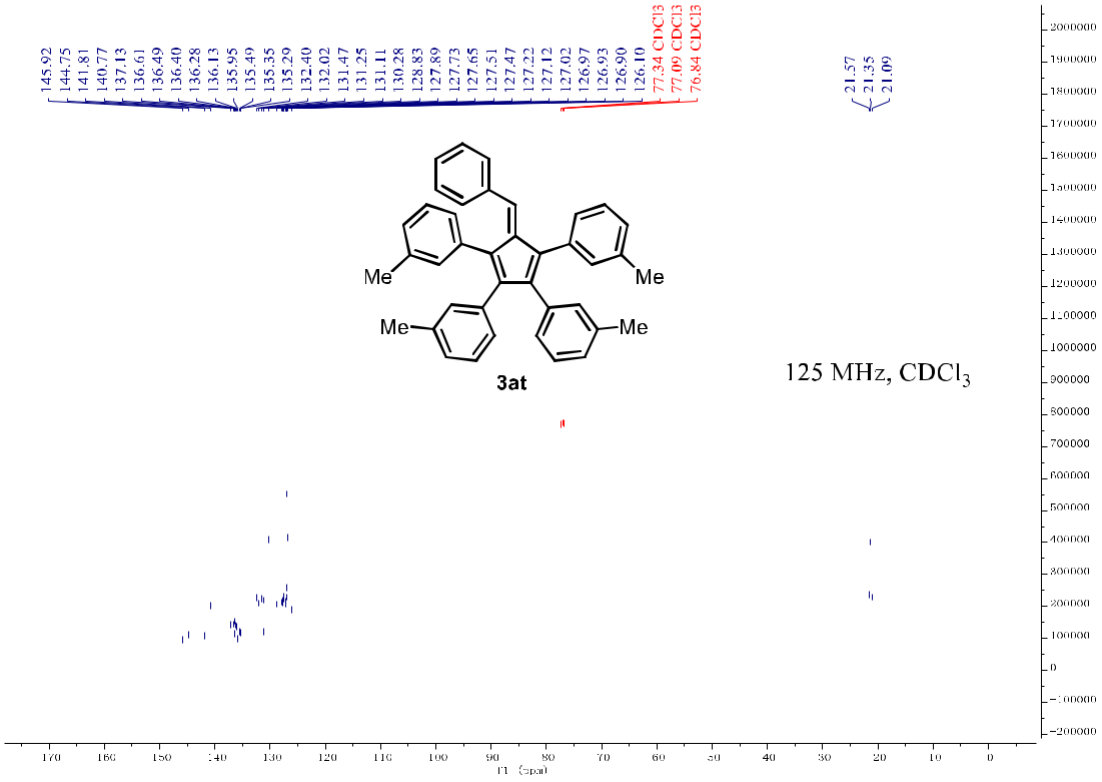
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methoxybenzene) (3as)



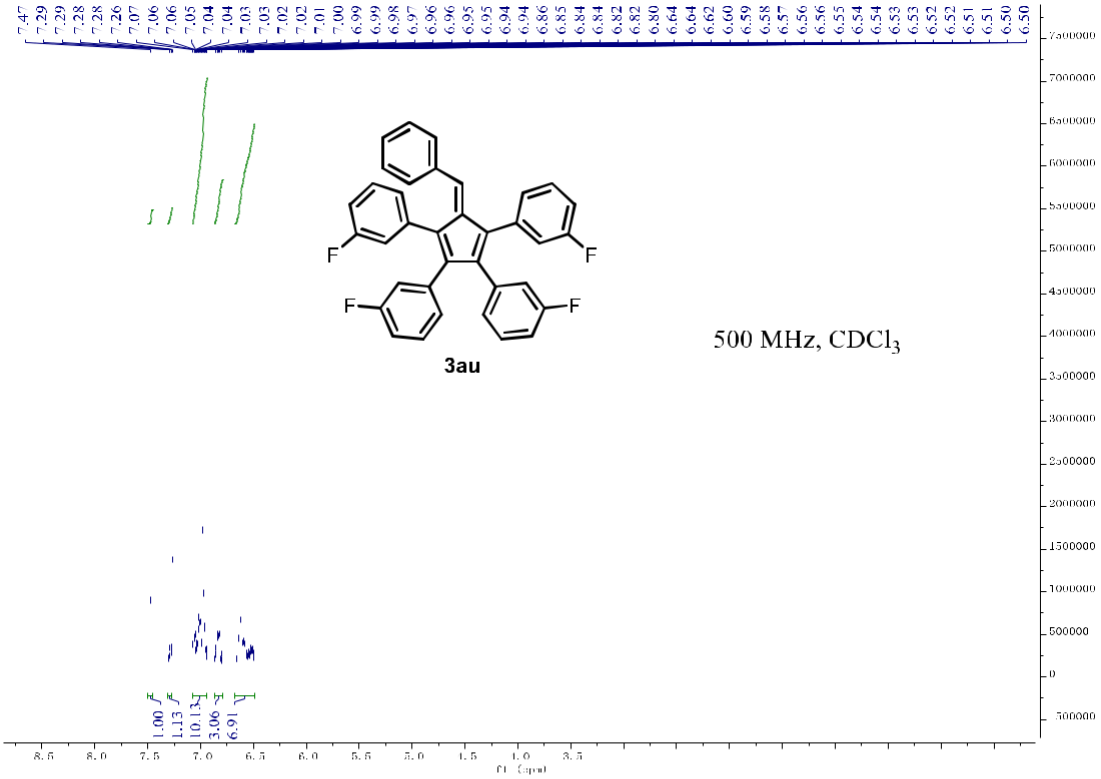


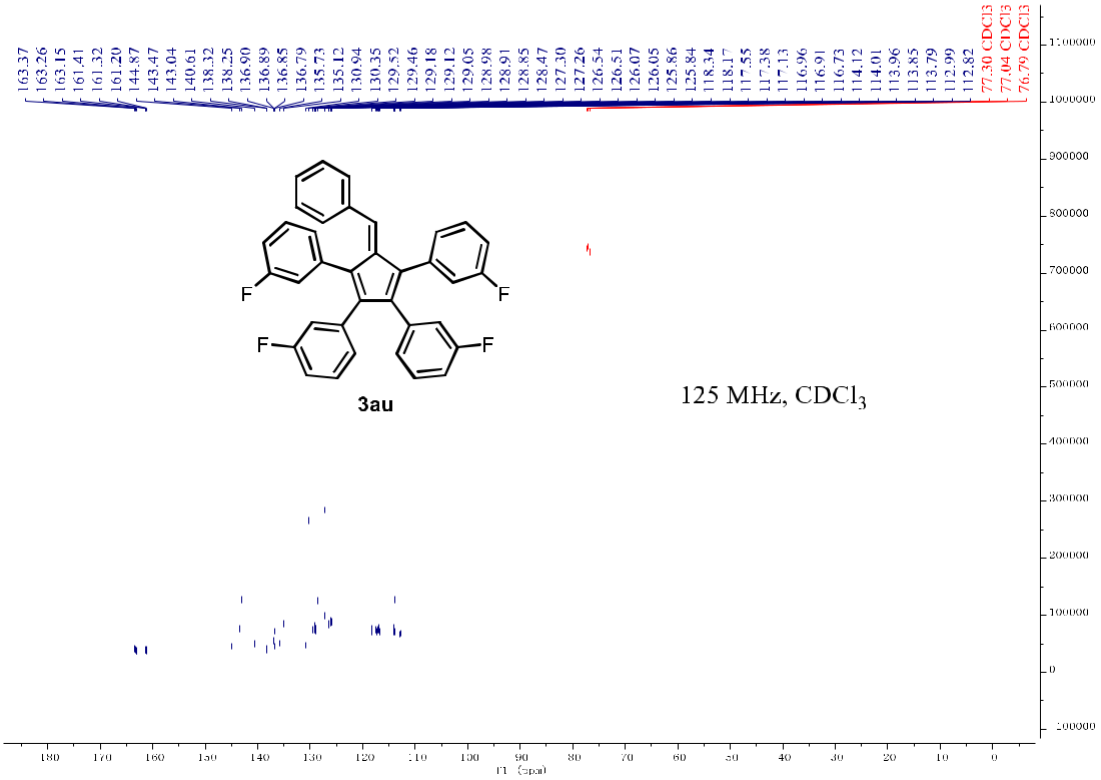
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(methylbenzene) (3at)



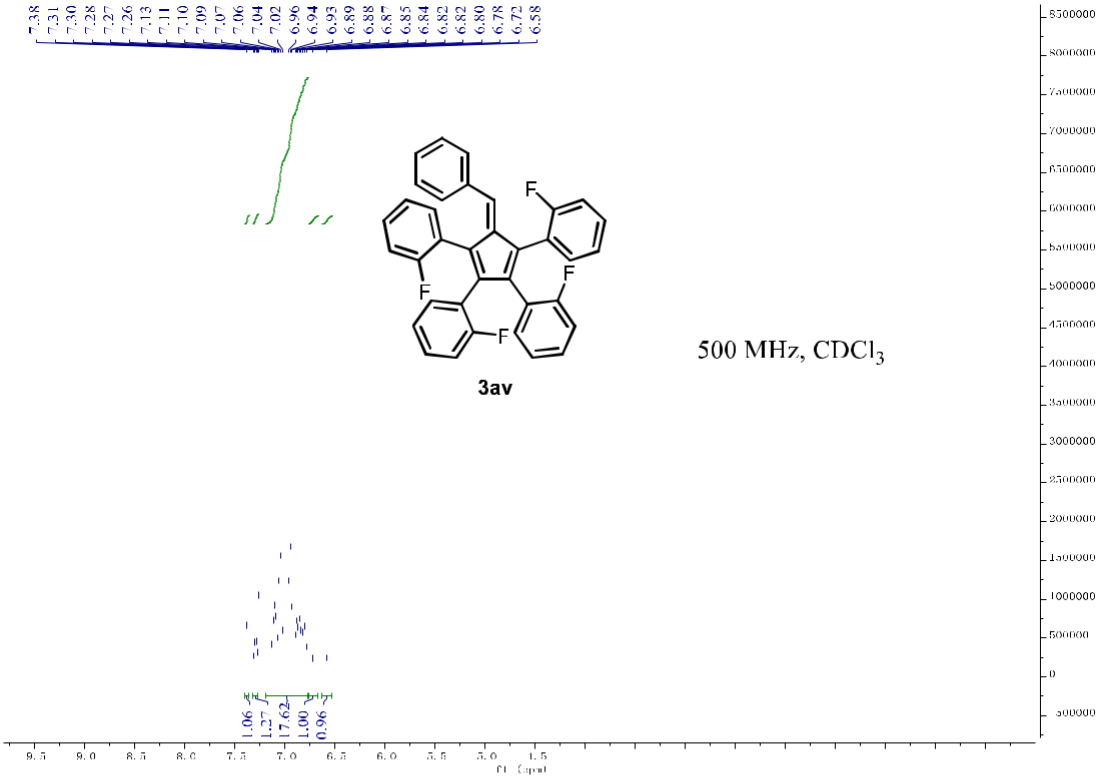


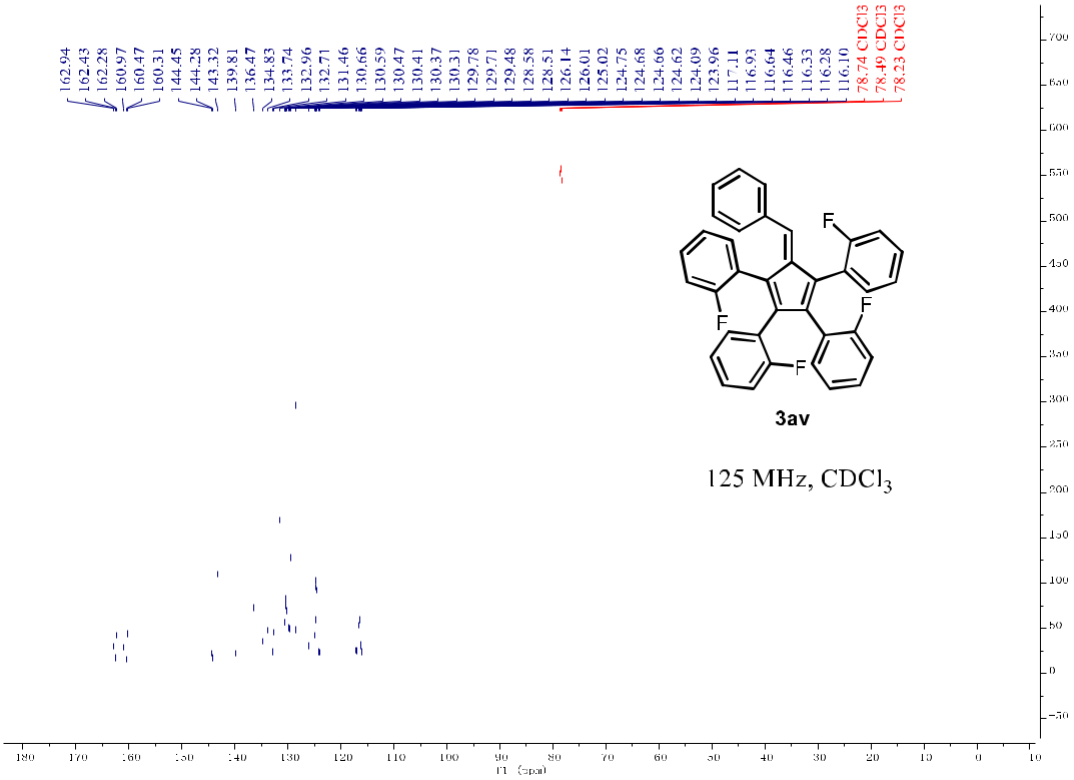
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene) (3au)



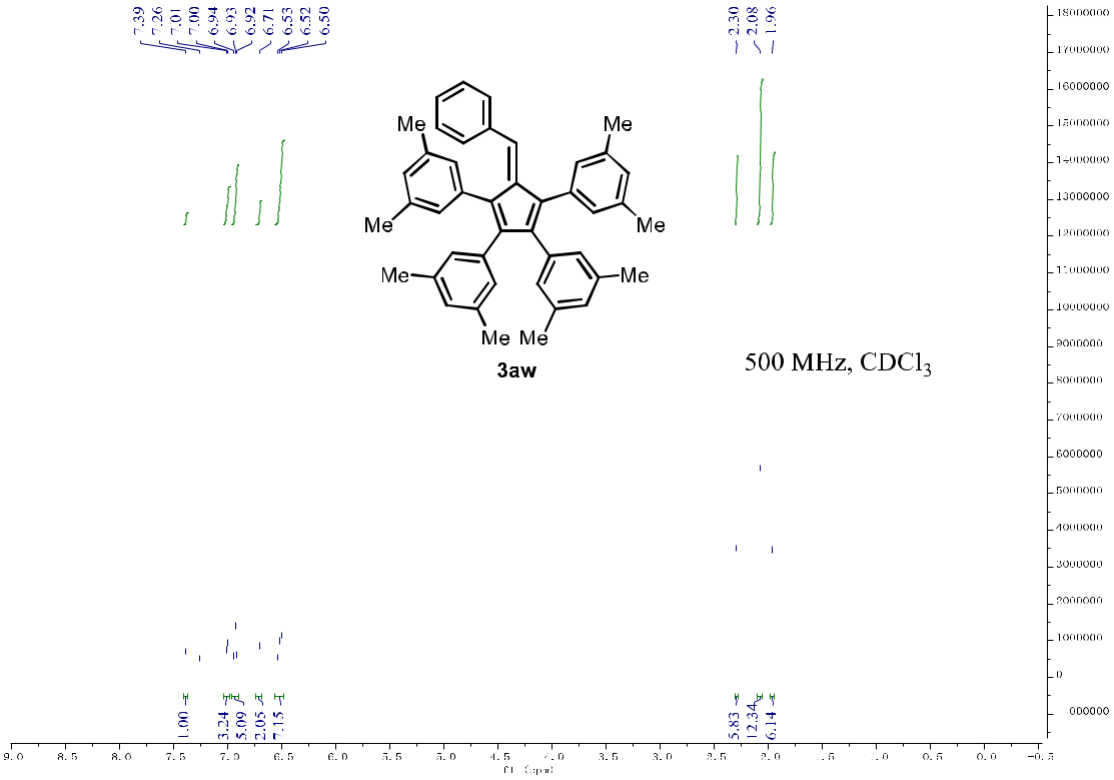


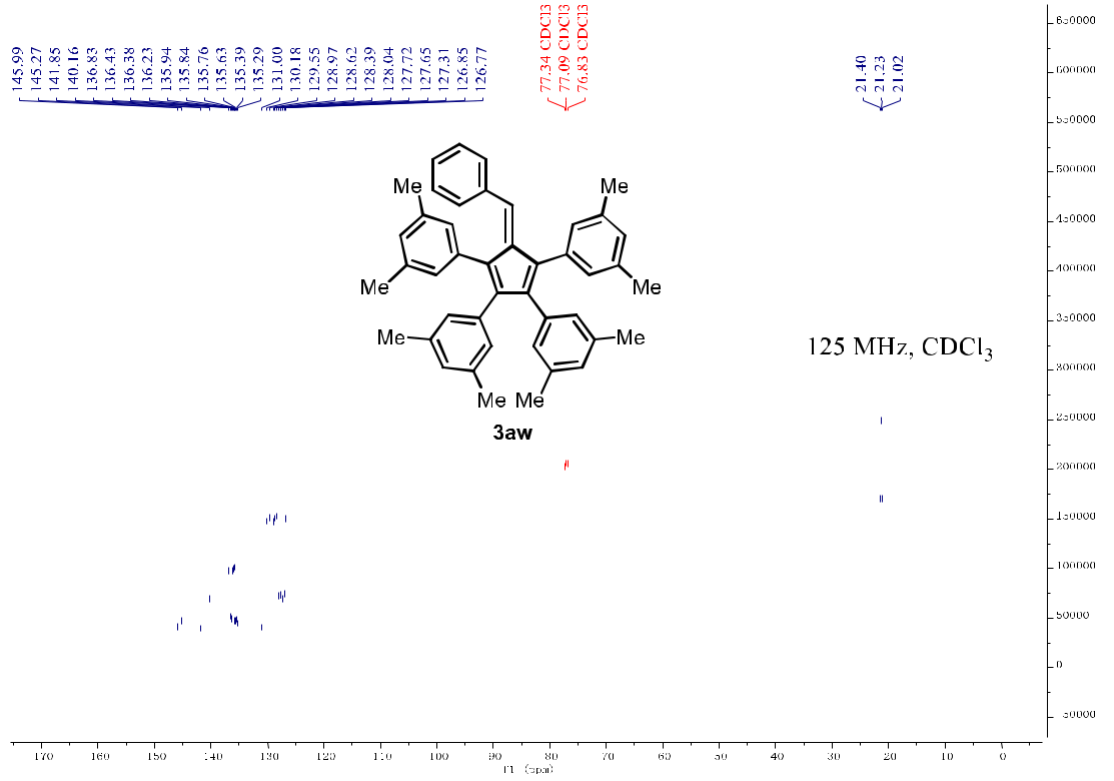
2,2',2'',2'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(fluorobenzene) (3av)



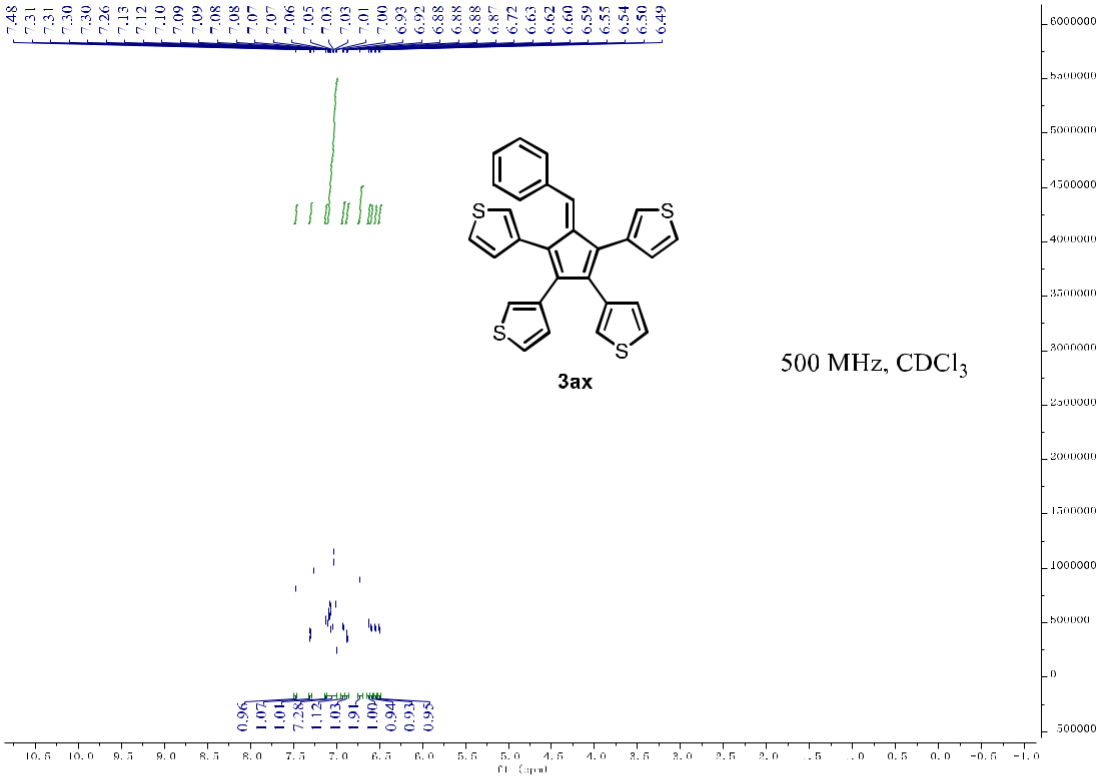


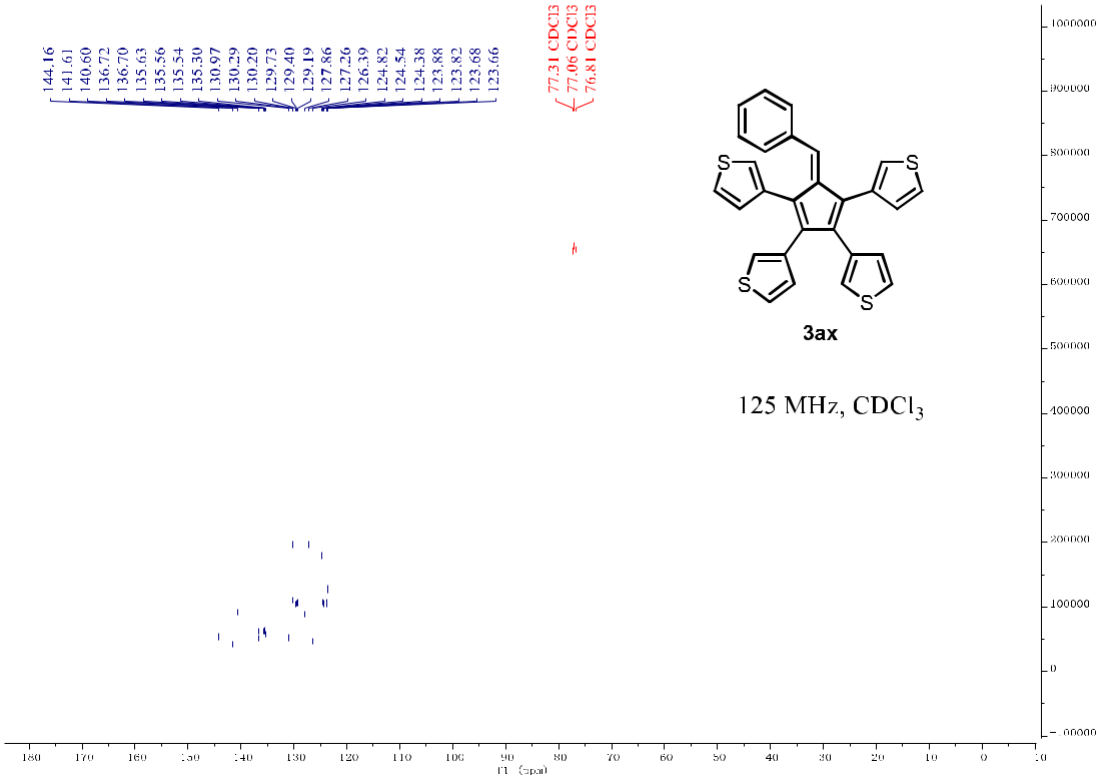
5,5',5'',5'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrakis(1,3-dimethylbenzene) (3aw)

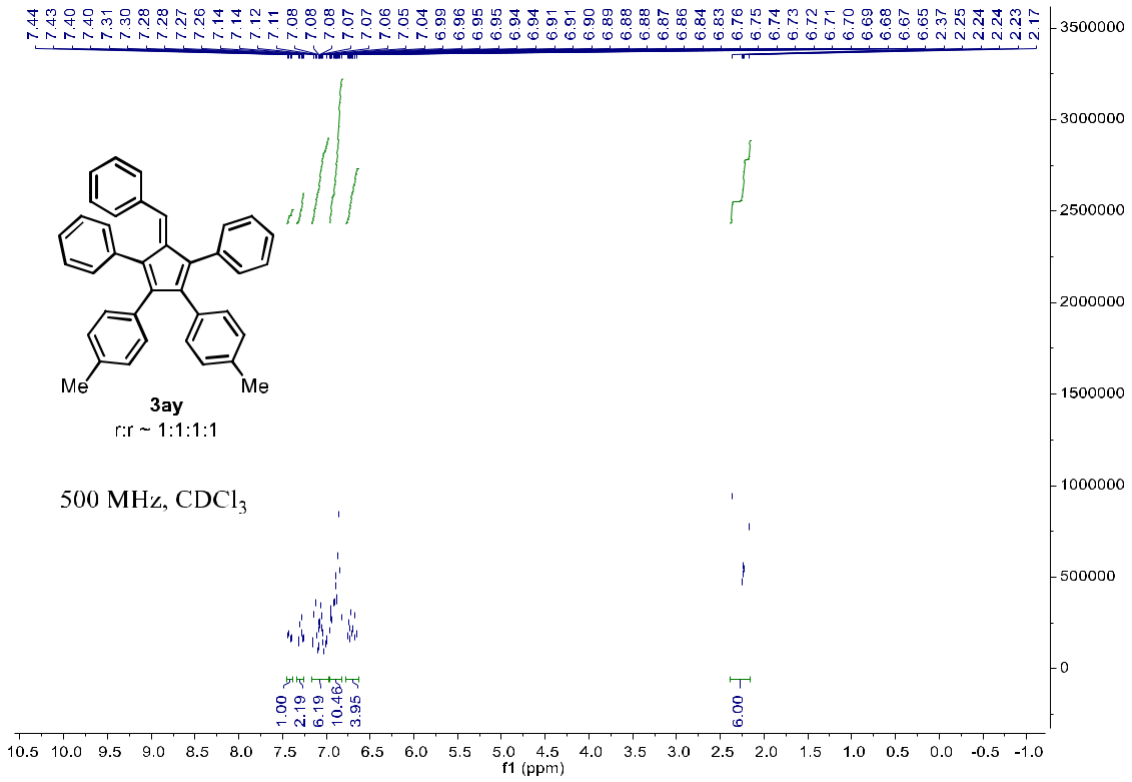


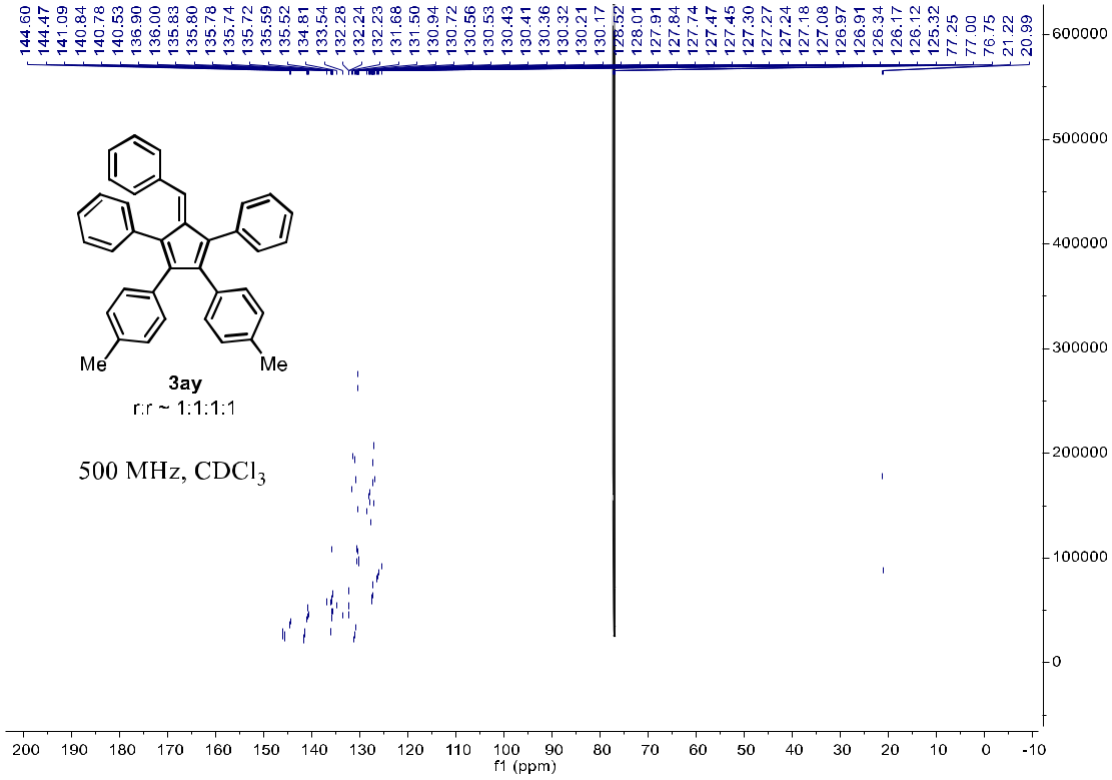


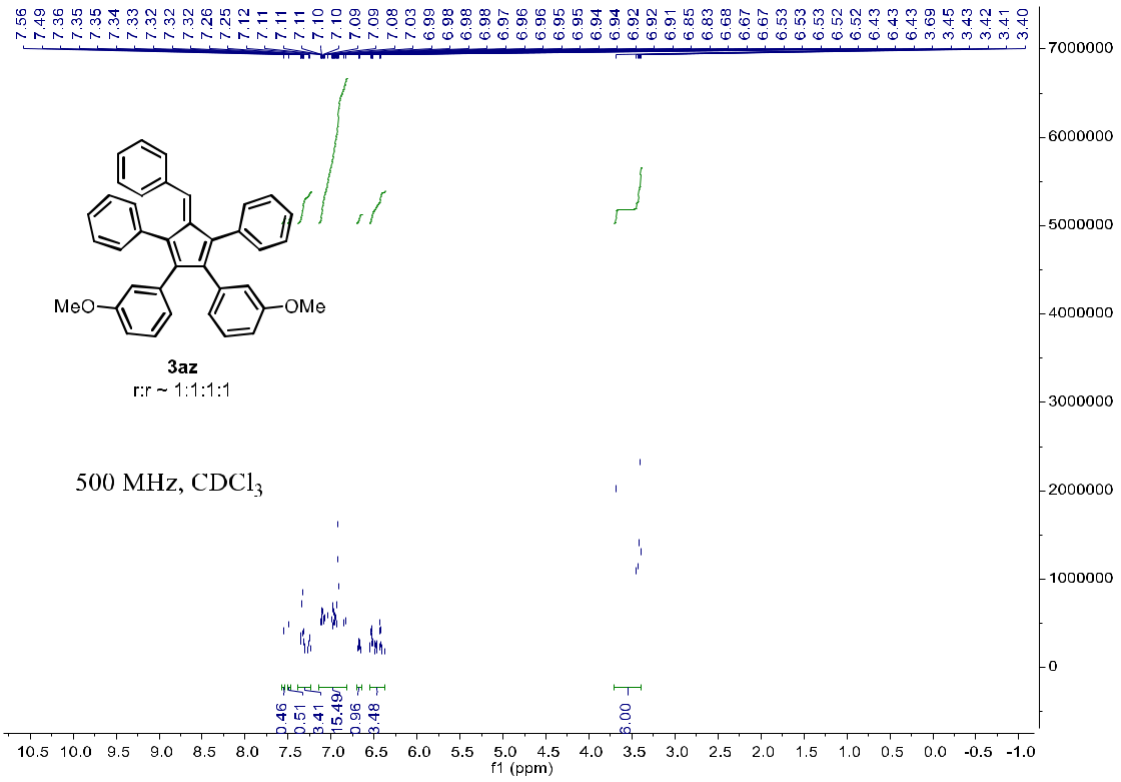
3,3',3'',3'''-(5-benzylidenecyclopenta-1,3-diene-1,2,3,4-tetrayl)tetrathiophene (3ax)

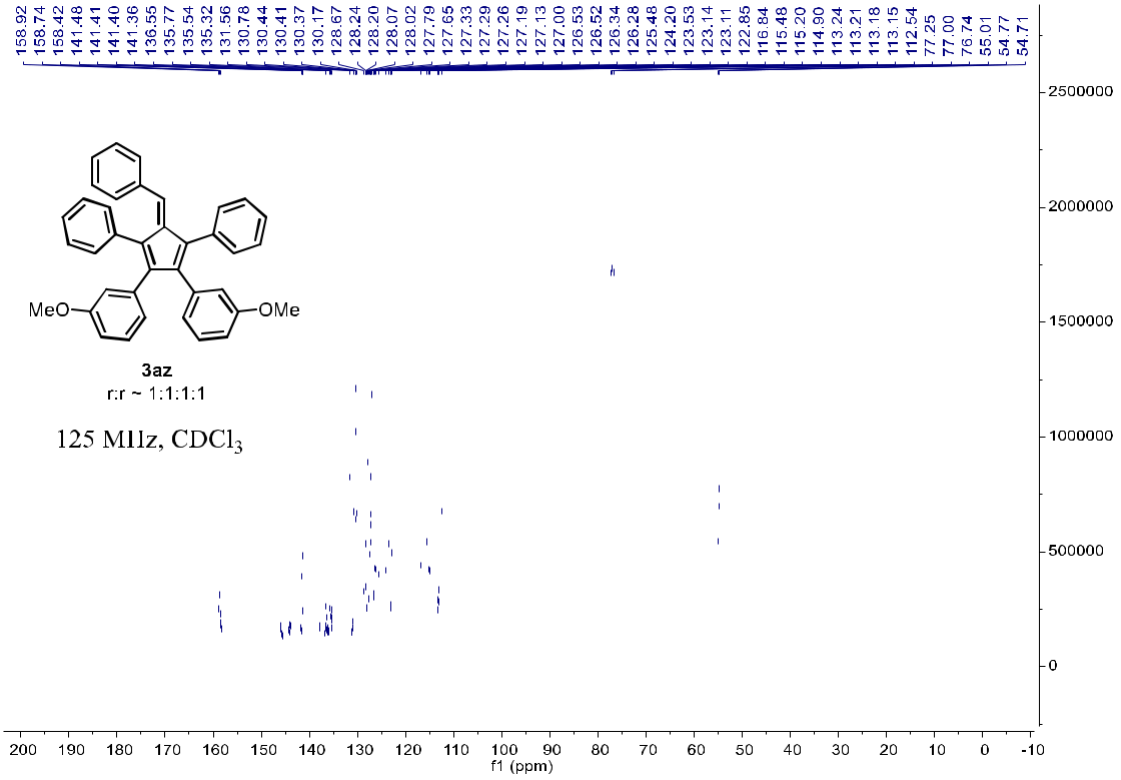


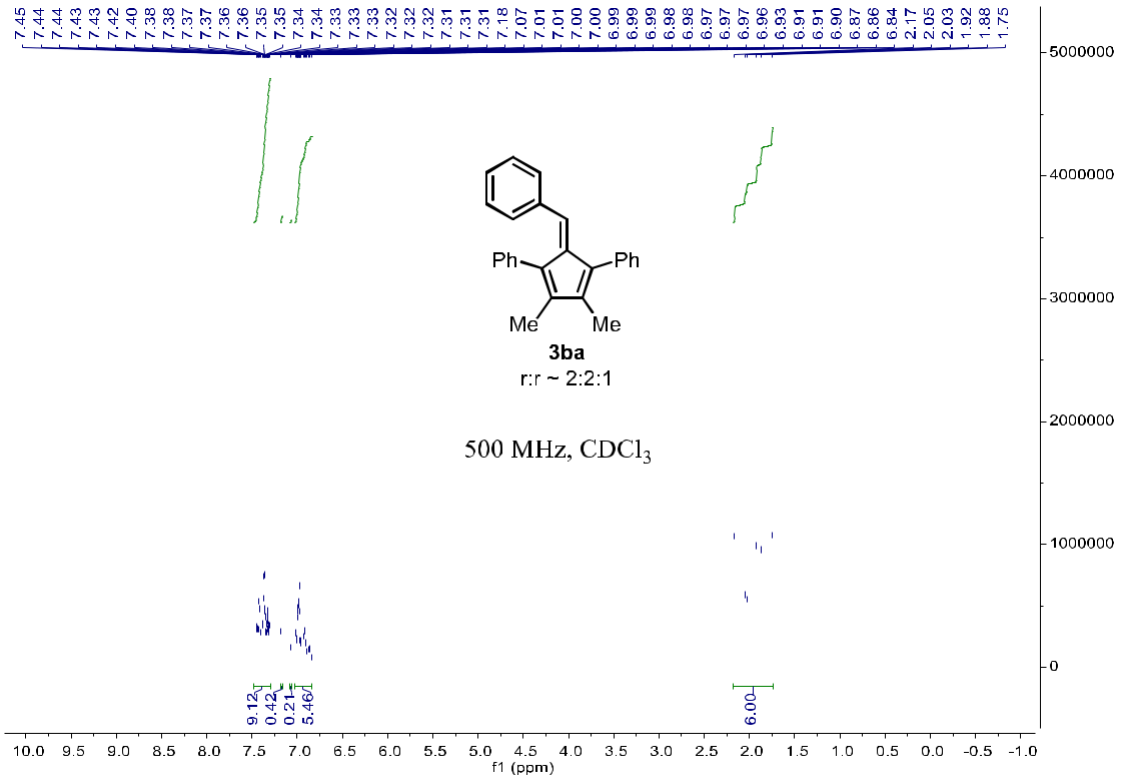


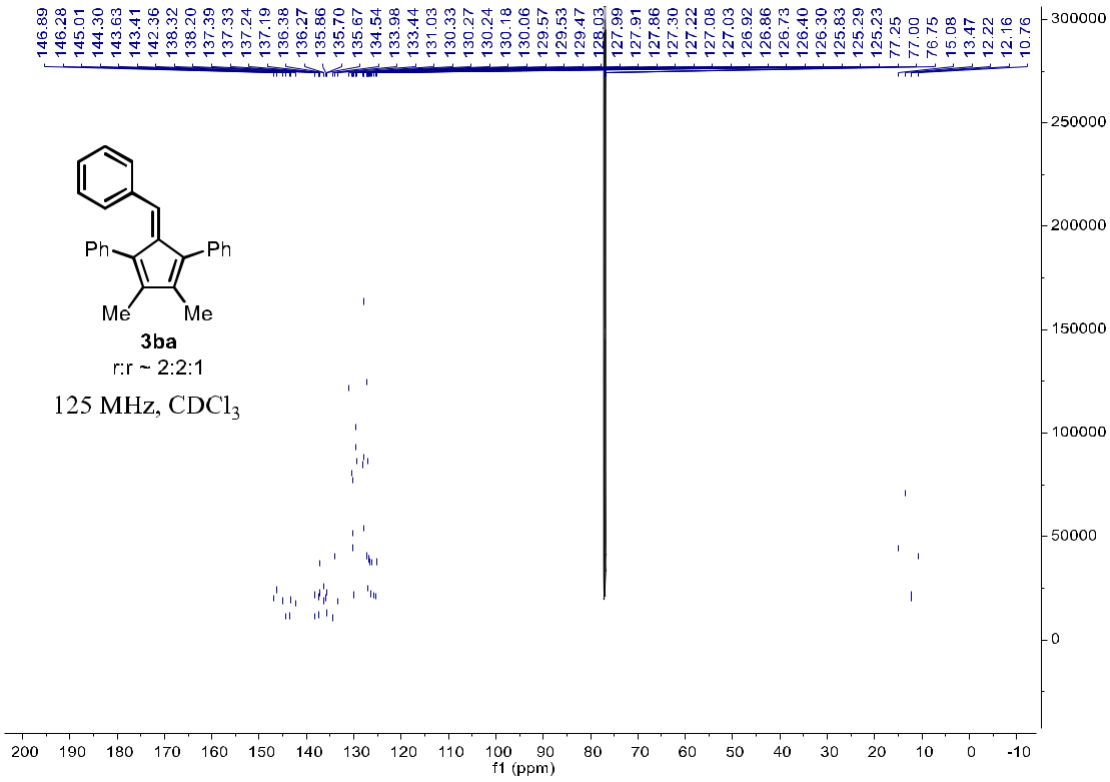

Me
Me
3ay
r:r ~ 1:1:1:1
500 MHz, CDCl3
f1 (ppm)


Me
Me
3ay
r:r ~ 1:1:1:1
500 MHz, CDCl3
f1 (ppm)

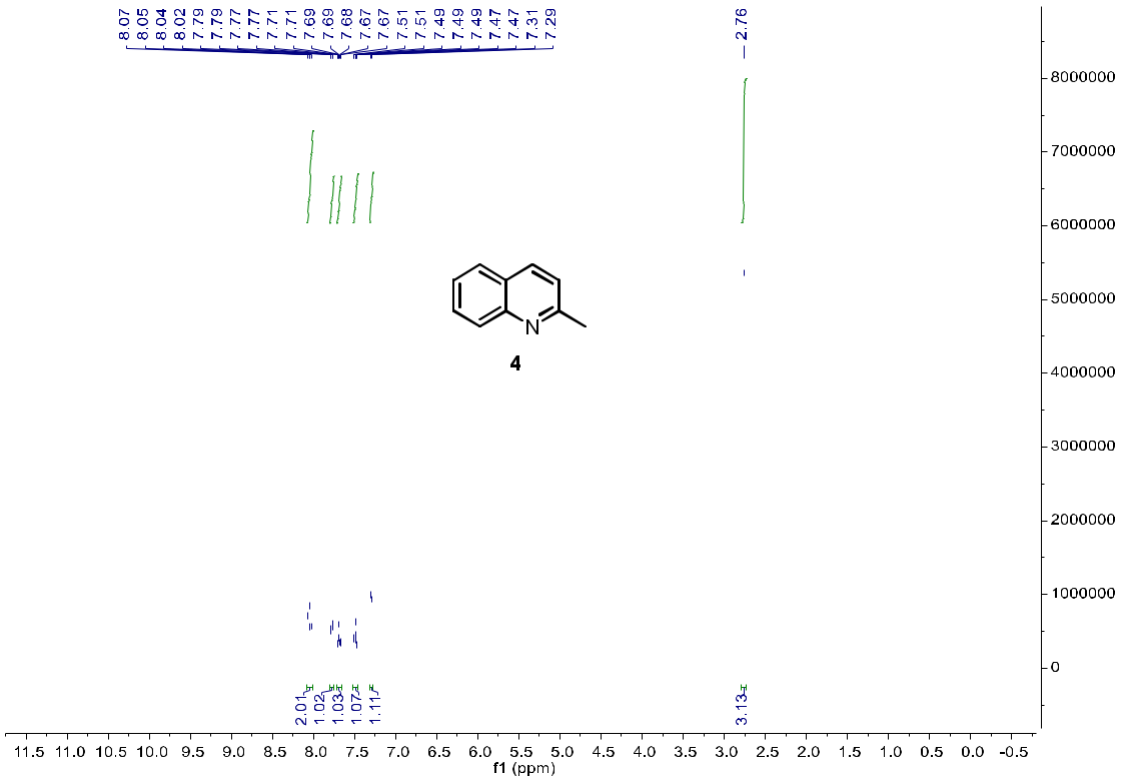

MeO
OMe
3az
r:r ~ 1:1:1:1
500 MHz, CDCl3
f1 (ppm)


MeO
OMe
3az
r:r ~ 1:1:1:1
125 MHz, CDCl3
f1 (ppm)





2-methylquinoline (4) ^{1}**H NMR** (400 MHz, $CDCl_3$)


8.07
8.05
8.04
8.02
7.79
7.79
7.77
7.77
7.71
7.71
7.69
7.69
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7.67
7.67
7.51
7.51
7.49
7.49
7.49
7.47
7.47
7.31
7.29
2.76
N
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1.02
1.03
1.07
1.11
3.13
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6000000
5000000
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3000000
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11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)